**Draft Minutes**

**Surface and Micro/Nano Analysis Working Group (SAWG) Meeting**

Hybrid Meeting on April 24-25th, 2023

Marie Curie Library (BIPM) and online

Draft Version 3, 5Jan2024

Attended by the following participants

In person:

Toshiyuki Fujimoto (NMIJ)

Alexander Shard (NPL)

Kyung Joong Kim (KRISS)

Michael Krumrey (PTB)

Seungmi Lee (KRISS)

Andrea Mario Rossi (INRIM)

Rainer Stosch (PTB)

Li-Lin Tay (NRC)

Donald Windover (NIST)

Online:

Ahmed Abou-Kandil (NIS)

Jariya Buajarern (NIMT)

Charles Clifford (NPL)

Norma Gonzalez Rojano (CENAM)

Werner Jordaan (NMISA)

Jeong Won Kim (KRISS)

Ansoon Kim (KRISS)

Hiroyuki Matsuzaki (NMIJ/AIST)

Caterina Minelli (NPL)  
Adlan Akram Mohamad Mazuki (NMIM)

Maria Mabel Puellel (INTI)?

Jorg Radnik (BAM)

Benjamen Reed (NPL)

Ali Enis Sadak (UME)

Rania Sayed (NIS)

Juliana Serna Saiz (INM Colombia)  
Egor Sobina (UNIIM)

Opening remark, appointment of rapporteur and participant introduction

The chairman of SAWG, Dr. Toshiyuki Fujimoto, welcomed everyone to the meeting and briefly stated SAWG scope.

* To carry out Key Comparisons, and where necessary pilot studies, to critically evaluate and benchmark NMI/DI claimed competences for measurement standards and capabilities for spatially resolved chemical surface analysis at the micro and nanoscale;
* To assist in identifying and establishing inter-laboratory work to improve the traceability of spatially resolved chemical surface analysis at the micro and nanoscale.

Chair appointed Li-Lin Tay (NRC) to be the rapporteur of the meeting. Chair asked if there are additional items to add to the agenda. None arise. Draft Agenda R04 is adopted.

Draft minute from the 2022 SAWG virtual meeting was posted online in June 13th, 2022. No comments were received as of the Apr. 23rd, 2023; therefore, draft minute was accepted as the 20th SAWG meeting record.

Dr. Fujimoto noted three major events happened since the 20th SAWG meeting.

1. CCQM K-157, “Measurement of the amount of substance of HfO2 expressed as the thickness of nm films” Pilot lab (Dr. Kyung Joong Kim) circulated a draft-B report on 12 June 2022 and a final revision on 22 December 2022. The final version included a statement on the traceability of each NMI’s measurements. The final report of the comparison was uploaded to the KCDB on 30th March 2023. 8 NMIs and 1 DI participated in the comparison and 8 countries were reported their results within the due date. Measurement methods used were XPS, XRR, TEM and MEIS. The average degrees of equivalence uncertainties for the HfO2 films with nominal thickness from 0.7 nm to 6.0 nm were in the range from 0.026 nm to 0.099 nm, respectively. It is anticipated that most of the participating laboratories will submit a CMC claim.
2. CCQM K-172, “Measurement of Specific Adsorption A [mol/kg] of Ar on zeolite at liquid argon temperature” Specific argon adsorption to microporous substances was complete in the previous period. Since the 20th meeting, ten CMCs were claimed by SAWG members using this key comparison as evidence. The space of measurement covered by the specific gas adsorption measurement comprises microporous (D<~2 nm) materials which are characterized by high values of BET specific surface area (500-1500 m2/g). CCQM-K172 was organized for microporous materials (D<~2 nm) to demonstrate CMC claims for gas adsorption method. Five of the CMCs claimed were for activated nanoporous carbon rather than zeolite.
3. CCQM-K166, “Measurement of nanoparticle number concentration in liquid suspension”

Key comparison K166 (number concentration of 60 nm gold particles) and P210 (mixture of 60 nm and 30 nm gold nanoparticles) commenced in January 2023. Dr. Heidi Goenaga Infante, project leader, noted in March 2023 that the samples were insufficiently stable, and these activities are paused until after the IAWG/SAWG joint meeting on this topic on 25th April 2023.

Participants (both online and in-person) are then invited to do a 3-minute introduction of themselves and their activities / techniques that are within the scope of SAWG.

* Li-Lin Tay (NRC-Canada) gave a brief introduction of the NRC measurement competencies that falls within the scope of SAWG. These includes the XPS, Auger, nano-Auger, SIMS, full suite of electron microscopy (SEM, TEM operates in vacuum and variable pressure) with EDX and EELS capability with focused ion beam for semiconductor sample preparation. Her research interest is in optical spectroscopy and microscopy and introduced the broad suite of optical spectroscopy and microscopy capabilities in NRC: MicroRaman, micro-FTIR both with chemical mapping and imaging capabilities, microspectrophotometer and micro-fluorimeter. Li-Lin is also leading the Raman task group project within SAWG.
* Juliana Serna (INM Colombia). Materials analysis group is part created in 2021 which addresses nanomaterials needs in Colombia. Relevant techniques in the team includes spICPMS and UV-Vis for NP size determination.
* Ahmed Abou-Kandil (NIS, Egypt): Introduce National institute of Standard in Egypt chemical metrology division. It covers references materials and analysis of food. For surface analysis, FTIR and Raman spectroscopies are among the capabilities planned.
* Jariya Buajarn (NIMT, Thailand): In surface analysis, it is a collaboration between physical metrology and chemical/biology metrology departments in NIMT. Instruments relevant to SA includes BET analysis and electron microscope and is a participant in nanoparticle concentration study with spICPMS.
* Alex Shard, Charles Clifford, Caterina Minelli and Benjamen Reed (NPL, UK): Dr. Shard gave a brief introduction on SA related activities. Capabilities includes XPS, SIMS, scanning probe and electron microscopies, has strong interest in nanoparticles, nanomaterials including graphene. Therefore, NPL has strong interests in Raman microscopy as well as measurement capabilities in organic and inorganic nanoparticles. NPL leads a number of sub-committees in ISO TC 201 and 229 & VAMAS technical committees.
* Norma Gonzalez (CENAM, Mexico): In the Emerging technology directorate the team focused in advanced materials (nanomaterials). Capabilities relevant to surface analysis includes SEM, scanning probe techniques, FTIR, Raman and XRF. Also has access to XPS and TEM from collaborators.
* Werner Jordaan (NMISA, South Africa): The materials characterization session covers surface analysis, structure analysis and advanced materials characterization. Relevant instrumentation includes TOF/SIMs, XPS, FIB-SEM, EDS, EBS, XRD, PSD. All of the instrumentations are quite new. The materials characterization is focused on support industries, provision of services to industries (petroleum and mining, nanotechnology, automotive, plastics, food sectors, packaging, electronics, air pollution particle sizing measurements..etc.); Also support academic researches.
* Kyung Joong Kim, Jeong Won Kim, Seungmi Lee, Ansoo Kim (KRISS, South Korea): There are a number of expertise in KRISS supporting surface analysis, including SIMS, MIES, XPS, FTIR, Raman spectroscopy. The team is focused on industrial metrology, e.g. thickness measurements and quantitively surface analysis. Dr. Lee is focused on XPS and Drs. Kyung Joong kim and Jeong Won kim are focused on advanced device system.
* Hiroyuki Matsuzaki (NMISJ/AIST, Japan) NMIJ has surface analysis capabilities in XPS, XRR, electron microscopies; Dr. Matsuzaki has expertise in ultrafast optical spectroscopy and is interested in the update on the Raman task group.
* Adlan Matzuki (NMIM, Malaysia): NMIM has capabilities in XRF analysis for Au purity, metal impurity in consumer product. NMIM also support R&D in consumer products analysis including metal contents in consumer products.
* Jorg Radnik (BAM, Germany): Dr. Radnik shared capability from the Surfaces analysis and interfacial chemistry group which covers nanosafety, nanomaterials, nanotechnology and nano-characterization, nano-data. Its activities cover three work packages including 2D materials, core-shell NPs, micro-nano-plastics, nanoCRMs, nano Reference data, standards and guidance. The work-package 2 is on Thin-film analysis and deposition with capabilities in Ellipsometry, MeOx layered system, semiconductors, porous structures, polymer membranes, modelling. In work package 3 covers energy materials, Hydrogen materials and concrete materials. He briefly introduced capability in TOF-SIMS for energy materials analysis and concrete. Hard energy XPS (HAXPS) has been applied to qualitative and quantitative chemical analysis mostly on the core-shell NPs, 2D materials and thin-films. Ellipsometry is used mostly for defect analysis on compound semiconductors for power electronics, phase transition measurements; FTIR and Scanning white light interference microscopy (WLIM) for film thickness analysis; High resolution SEM/EDS for NP speciation; PVD-E-beam evaporation. The group is also very active in VAMAS committees.
* Ali Sadak (Tubitak UME, Turkey): Surface analysis is under the Organic chem group. Relevant capabilities in surface analysis includes: various SEM, TEM, AFM, micromeritics 3-Flex BET with 8 different gases.
* Rania Sayed (NIS, Egypt): Focused on characterization of nanomaterials, XRD, TEM, SEM, AFM. For surface analysis, interested in Raman and other optical techniques and has access to other collaborators.
* Michael Krumrey and Rainer Stosch (PTB, Germany): The PTB Berlin facility has activities with synchrotron radiation related to surface analysis including: XRR, XRF, NEXAFS (Near edge X-ray fluorescence structure spectroscopy), Grazing incidence X-ray fluorescence analysis, Grazing incidence small-angle x-ray scattering, small-angle x-ray scattering (SAXS). Many of these techniques have been used to participate in many of the SAWG key comparisons. Dr. Rainer Stoch introduced the capability of general organic chemistry at PTB Braunschweig. The group covers surface analysis including chemical composition for solid surfaces which uses laser ablation coupled to ICPMS with traceability through isotope dilution approach; Confocal microRaman spectroscopy in 2D and 3D mapping with SPM support experimental work with software for 2D mapping and simulations in DFT; dimensional calibration Raman standard for 2D mapping (traceable); Raman spectral calibration for strain and stress measurement with high Raman shift accuracy; it also support other PTB division on 2D materials development.
* Andrea Rossi (INRIM, Italy): Physical chemistry and nanotechnology with focus on food metrology with recent focus on food packaging and particularly on the reduction of plastic use. New materials as alternative food packaging by modify surface of polymer to have anti-bacterial properties for food waste reduction and reduction; Has expertise in hyphenated Raman spectroscopy particularly for microplastics study including field flow fraction coupled to Raman for speciation of plastics for Food safety and drinking water safety application. Class II laboratory capability with wide suite of vibrational spectroscopy capabilities. The team coordinated the EMN’s safe and sustainable food project.
* Donald Windover (NIST, USA): NIST has a large number of SRM covering large areas; many of the SRM are relevant to surface analysis. Capabilities includes XRD, XRF, X-ray reflectivity. Reference materials relevant to this communities includes strain reference materials of thin single layer structure, SAX with high energy transmission through wafer. Another reference materials under discussion is the ion-implanted materials. NIST neutron facility has capability for ion-implantation measurement and is currently under-development. More update will be shared in the next year or so.
* Egor Sobina (UNIIM, Russia): Dr. Sobina is the director of UNIIM and has expertise in CRM of porous materials for gas adsorption methods.

Brief report of the KCWG meeting / A Shard(NPL)

The KCWG meets throughout the year and the last meeting was April 18th, 2023. Since the 20th SAWG meeting, there are 10 SAWG CMCs submitted and approved. All were submitted by Tubitak, related to absorption and BET surface area, related to Ar absorption and BET surface area for zeolite and with the “how far the light shine” extend to nanoprous carbon. Dr. Shard thank all SAWG members who helped with the review of the CMCs. He also urges the SAWG member to think how future broad scope claim can be done.

KCWG paper on metrological traceability is currently under discussion. Under CIPM-MRA-P-11 states that metrological traceability of participants to CIPM MRA via one of the following two routes: Primary realization or through another participant having relevant CMCs with appropriate uncertainty published in KCDB or through calibration and measurement services offered by the BIPM. How does it apply in terms of broad scope claims (especially through the second route); e.g. newer area (such as SAWG), metrological traceability is not always straight forward. A general observation of the recent developments in broad scope CMCs, the KCWG have noticed it is becoming more difficult for NMIs claiming metrological traceability through the second route.

Recommendations from KCWG. (a). In Chem-Bio metrological traceability via route 2 could also be established through another participant having demonstrated measurement capability for the unit of measurement and the defined measurand concerned with appropriate measurement uncertainty through multiple successful participation in recent CIPM key comparisons, RMO key comparisons and supplementary comparisons. Traceability through route 1 includes institutes using CRMs or high-purity primary chemical reference materials that have been value-assigned by applying their own measurement capabilities recognized within published CMCs or via demonstrated measurement capability for the unit of measurement and the defined measurand concerned with appropriate measurement uncertainty through participation in CIPM key comparisons, RMO key comparisons and supplementary comparisons.

It is up to the working group to decide on how to approach this and examine each case as it arises. Alex welcomes any comments.

* A question from Dr. Krumrey (PTB) regarding the CMCs for HfO2 thin film study? Can scope be broader than HfO2?

Dr. Shard responded: For now, HfO2 film thickness is all can be claimed, but in future, it is possible to extend the measurement traceability to extend to other thin-film on top of wafer.

Experience at the work with KCDB2.0 / Egor Sobina(VNIIM)

Chair invited Dr. Egor Sobina to provide an overview in working with KCDB2.0.

Egor went through the step-by-step requirement for registration of new KC including starting by filing the registration form, which includes measurand, nominal values, pilot, participants, dates and contact person’s information. Followed by technical protocol, final report, result summary and publication of the final report in Metrologia’s technical supplement. He also described how to navigate the KCDB website to create and upload the final report.

Dr. Sobina also showed the working group participants on how to navigate KCDB2.0 in the creation of CMCs. This involves filling out various fields such as classification of services, measurands, expanded uncertainty and references. A preview window allows one to review all of the input information before the final submission.

Update of the Raman TG / L.-L. Tay(NRC)

Li-Lin Tay (NRC) presented a report on the Raman task group activities which included (1.) Progress on the quantitative measurements with Raman spectroscopy on mixed polymer films and (2.) Raman measurement related activities within VAMAS TWA 41 and 42 as well as the various activities from the ad-hoc international Raman metrology group.

1. On the traceable quantitative measurement of mixed polymer weight fraction with Raman spectroscopy, Li-Lin briefly recap the goal of the study. The aim is to determine the weight fraction of mixed polymer blend. Two miscible polymers, polystyrene (PS) and polypropylene oxide (PPO) of different weight fractions are prepared using traceable gravimetric technique with calibrated balance traceable to Kg. A set of 5 samples of known weight fractions are prepared to allow for an establishment of a calibration curve. Two blind samples are prepared for their weight fraction to be determined by the calibration curve.

Li-Lin noted that since the project was first introduced, her team at NRC has continue to work on improving the sample preparation of the thin polymer films. This includes trying out different solvent for the dissolution and deposition on welled- vs planar glass substrates. She shows some preliminary results of the calibration curve determined from a sample set and the test of two un-known samples.

She presented a brief flow-chart of proposed protocols which includes the sample preparation by pilot lab and their distribution to participant labs; measurement requirement from the participants. However, she noted that it is not certain if all data should be returned to the pilot lab for analysis or should the participant simply return the weight fraction value to the pilot lab which then prepares the comparison result. She presented a tentative schedule for the proposed pilot study with review of protocol by the international Raman metrology group by spring of 2023, potential bilateral through VAMAS TWA 42 project and the initiation of pilot in late 2023.

1. In the second part of the presentation, Dr. Tay gave a brief summary of the five on-going VAMAS TWA 41 and 42 Raman spectroscopy measurement projects. These included the following.

* TWA 41 Project 1 on “Raman measurements of CVD-Grown graphene” led by Andrew Pollard (NPL) and Ling-Ling Ren (NIM). The project was completed and published in 2D Mater. 9, 035010 (2022).
* TWA 41 Project 11 on “Disorder and number of layers of graphene flakes” co-led by Chiara Portesi (INRIM), Andrew Pollard (NPL) and Lingling Ren (NIM). Project started in Dec. 2022. The objective is to quantify the number of layers in graphene flakes with Raman spectroscopy. The results from the project will help to guide future revision of ISO/TS21356-1 “Structural Characterization of Graphene” within ISO TC229 JW2.
* TWA42 Project 2 on “Raman spectroscopy for TiO2 nanoparticles mixture” led by Andrea Rossi (INRIM). The goal is to use Raman spectroscopy for the phase quantification of TiO2 binary mixtures. Project had 12 participants. All returned data with good agreement of the % phase content. All measurements have been completed and analysis by the PL is also completed. The manuscript is ready for submission.
* TWA 42 project 4 on “Measurement of lateral resolution of Raman microscopy with nanowire artefacts” led by Sebastian Wood (NPL). Small diameter of InAs nanowires of ~ 110 nm grown across two metal pads are used to determine the lateral resolution of the microRaman measurements. There are total of 15 participants. Four sets of samples were sent around to 12 partners and as of April 2023, 9 sets of measurements results have been sent to the pilot lab for analysis. Because different partners are using different samples which all have slightly different InAs nanowire diameter, so that the comparison was done between the lateral resolution as identified by two nanowires on the same devices. Preliminary results show that most labs, indeed, reproduce the lateral resolution by two different nanowires on the same device. Only one participant has not being able to achieve this. Final evaluation will likely take place in the next few months once the remaining results are completed.
* TWA 42 project 5 on “Factors affecting reproducibility in Raman spectroscopy” co-led by Paul Finnie and Li-Lin Tay (NRC). This is a survey project aimed to reach out to a broader user community of Raman spectroscopy to understand the technical challenges and measurement reproducibility. The survey ran from Mar. 2021 to Dec. 2022. In total, 83 responses were received. Key findings from the survey indicates the following factors (and level of dis-satisfaction in brackets) are reported as technical challenges in reproducibility of Raman spectroscopy.
* Relative and absolute Raman intensity (48% and 60%)
* Raman Shift (18%)
* Confocal volume (56%)
* Focal spot size (52%)
* Stability of sample under laser exposure (38%)
* Data processing: Machine Learning (81%), chemometrics (67%), spectral matching or statistical analysis (50%)
* Other remarks: SERS (reproducibility, sensitivity, standard surface & molecule); users don’t know how to use correction/standards (e.g. intensity correction); power meter reproducibility; reproducible background subtraction
* The ad-hoc International Raman Metrology Group is a virtual meeting hosted by Dr. Angela Hight-Walker (NIST). It meets virtually once a month (1st Thursday of the month) and always try to organize an in-person meeting with a relevant conference once a year. Please reach out to Dr. Hight-Walker if anyone is interested in joining.

Questions and suggestions from participants:

* For the study, homogeneity of the mixed polymer film will need further improvement before the pilot study can be launched
* It was suggested that the intercomparison exercise be tested in VAMAS first. For VAMAS study, the protocol needs to be very specific to make sure the measurements and data analysis were carried out consistently by the participants.
* For the SAWG pilot, we can define the measurand but PL cannot dictate which methods participant uses to carry out the measurements.
* It is suggested that SAWG also reach out to OAWG to coordinate across WGs and make sure interested participants from both WGs can participate.

## Proposal: Pilot study on Elemental composition of an ionic liquid surface / Joerg Radnik(BAM)

Dr. Jorg Radnik (BAM) gave the presentation on the proposal of the pilot study on elemental composition of an ionic liquid surface. The samples proposed are salt with a melting point below 373 K which typically consisting of light elements (e.g. C, N, O, F, S, P, B, Cl, Fe). They are

Homogeneous in both lateral and vertical direction. They are stable in both UHV and X-ray therefore suitable for XPS and EDS. Dr. Radnik went through preliminary mini-interlaboratory comparison between 3 labs and the root mean squared deviation from stoichiometry using Peak, Touggaard and background are 0.64%, 0.68% and 0.60% respectively. The results show the ionic liquid system is well suited as reference for quantification of XPS and XRF for light elements.

The next step is sought participants for the pilot study. The proposed ILC will have 2 or 3 samples of ionic liquids and protocol ready for Oct. 2023. Anticipated results from participants in 01/24 and preliminary results presented at the next SAWG meeting in 04/24.

Questions and comments from participants:

* Dr. Krumrey (PTB) enquired about XRF results for the ionic liquid pilots. Dr. Radnik replied that there is no XRF results yet. Dr. Krumrey note that the XRF is applicable and the result would be interesting and PTB is interested in doing the XRF measurement of ionic liquid.
* Dr. Kim raise the point of if a CRM is needed to perform the intercomparison. Dr. Shard noted that the XPS does not need a reference material, just need to make sure the spectrometer itself is well calibrated. The stoichiometric value, ionic radii of the molecule is used as the reference value. Dr. Fujimoto asked about the certification of the stochiometric value and purity of the ionic liquid. Dr. Radnik indicated the purity is determined by the fact that XPS didn’t observe any other elements. It was then suggested that another independent method should be used to verify the purity of the ionic liquid, e.g. ICP-MS? Or a chromatography-MS technique can be sued to verify purity. Dr. Shard noted that for a pilot study, the purity of the material may not be a problem.
* Dr. K Krumrey further enquired if ionic liquid is a liquid. Dr. Fujimoto confirmed yes, and it has no vapour pressure. Using fundamental physical properties, its composition can be determined with good accuracy. It is noted that uncertainty of the physical properties needs to be taken into account and some of these components can have significant uncertainty.
* Chair call for potentially interested participants. The following participants indicated either interest or need to consult with expert in their institute. NPL, BAM, PTB, KRISS, NMIJ (maybe), NIST (maybe), NMISA (maybe)
* Chair will share the BAM protocol and measurands to SAWG for review. Any interested participants can let the chair know of their interest to participate. In the meantime, registration for pilot can be initiated.
* It was also suggested to reach out to inorganic analysis working group to seek potentially interested participants.
* Dr. Werner Jorddaan enquired about the sample’s compatibility to vacuum. Dr. Shard answered yes and the traceability can be to NPL Cu, Ag and Au. Dr. Werner asked if the spectrometer calibration information can be shared for evaluation. Dr. Shard will send Dr. Jorddaan the references for instrument calibration.
* Chair confirmed the measurands to be the elemental composition of C, N, O, F, S, P (B, Cl, Fe) (mole fraction excluding Hydrogen).
* Dr. Windover (NIST) enquired if the pilot will accept participant who can only cover partial list of measurands. Dr. Shard confirmed yes.

Proposal: Pilot study for the quantitative analysis of alloy films by multiple point calibration method / KJ Kim(KRISS)

Drs. Kim and Lee (KRISS) presented a new proposal for a pilot study on the quantitative analysis of alloyed films using multi-point calibration methods.

Many different surface analytical techniques (e.g. SIMS, XPS, AES, MEIS, RBS, TEM and SEM) can be applied to the quantification of major elements. Dr. Kim recap the 1998 VAMAS study of the Co-Ni and Au-Cu alloy. In the case of the Co-Ni alloy study, the system was ideal with no sputtering nor matrix effect. However, for the Au-Cu alloy system, severe matrix effect was observed. Previous CCQM SAWG key and pilot comparisons on Fe-Ni alloy films (2018, P108 and K67) as well as the CIGS films (2016, K129) to determine the mole fraction were briefly summarized. In K67, the sample consists of two elements with similar mass and the participants observed a small matrix and sputtering effect. However, in the case of K129, the sample is polycrystalline, contains 4 elements of different masses. Sever matrix and sputtering effect were observed.

In terms of traceability, isotope-dilution ICP/MS (ID-ICP/MS) can be used. Dr. Kim showed that for 100 nm thick AuxCu1-x and PtxNi1-x film, RBS results are within 1% of the ID-ICP/MS results. He also showed the second independent methods for traceability through ICP-AES. Results from these two techniques shows good agreement.

He then discussed the severe distortion of the composition analysis due to matrix and sputtering effect by XPS. He presented corrections that can be applied to mitigate both effects in the alloy reference. He recap the P-140 study of the Cu(In, Ga)Se2 film with the proposed correction factors applied to compensate both effects. Dr. Kim then discussed fabrication of the reference PtxNi1-x samples and preliminary RBS and XPS analysis of the reference film. He showed the XPS data fits well with 5-point quadratic curve and proposed this can be used to build the calibration curve. Measurement protocol for the pilot study were presented. The measurand is mole fraction (% mol/mol) of Pt and Ni in PtxNi1-x films. Samples will consist of 2 pure metal films (Pt and Ni), 5 reference (PtxNi1-x) films (x = 0.2, 0.35, 0.50 0.65, 0.80) and 1 unknown test specimen. Proposed measurement methods are XPS, AES, MEIS, RBS, XRF, EPMA and other available techniques. Participant should submit the mole fraction (% mol/mol) and uncertainty. Proposed comparison timeline is to circulate protocol in 06/23, pilot begins in 07/23 with results ready to be presented by 04/24 and immediately follow by the discussion for a KC.

Questions from participants:

* Dr. Krumrey enquired about the film thickness and substrate for which the PtNi film will be deposited. Dr. Kim confirmed it will be 100 nm of PtNi film deposited on Si substrate.
* Dr. Rossi asked about the dynamic range of the composition and if the response extends to 100% composition. Dr. Kim clarify that the current sample set will cover the composition range from 40-90%.
* Dr. Shard noticed the quadratic plot does not go through origin. Dr. Kim noted the presented quadratic plot is only valid in the range specified by the protocol which ranges from 40 – 90%.
* And Dr. Krumrey seek clarification of the x value in the PtxNi1-x sample description. He noted that the x value shown in the slides were very different from the composition determined by the XPS (e.g. for sample Pt0.2Ni0.8, the XPS analysis showed 40% of x but RBS showed a 25.8% x values). He wonders what would x-value be when analyzed with XRF; and if the same quadratic calibration curve be used in the case of XRF. Dr. Kim replied that the x value is the deposition target and should not be used as an indication of the true x value. He further noted that the XPS is plagued by both sputtering and matrix effect, however, both effects are minimized in the case of XRF measurements. The x-value as determined by other methods can be compared to RBS results which is within 1% of the ICP/MS determined value.
* Dr. Shard enquired about the traceability and if the samples based on the 7 reference materials will need to traceable back to KRISS. Dr. Krumrey also asked if the traceability should go to the sample or to the measurement methods? He suggested that the method itself be traceable rather than through the set of reference samples. He further suggested to remove the word “reference” from the reference samples. A few other participants also agreed that the word reference should be removed. Dr. Kim and Lee agreed.
* Most of the participants also agreed to remove the term “reference” from the “5 reference PtxNi1-x films”
* Dr. Fujimoto suggested that the molar ration be used so that other contaminants on the surface will not be called into question. He also enquired about the film purity, to which, Dr. Kim noted the 99.5% film purity.
* Potential participants include KRISS, PTB, BAM and the following NMIs needs to check-in and will confirm via email: NPL, NMIJ and NIST.

Proposal: Follow up comparison of the K-136 / Ali Enis Sadak(TÜBİTAK) and Egor Sobina(VNIIM)

* Tubitak (Dr. Sadak) and VNIIN (Egor Sobina) presented a proposal for a new KC as a follow-up on the K-136 of Mesoporous solids with pore size 2 – 100 nm); S(BET) range between 1 – 1000 m2/g. In K-136, Al2O3 is the adsorbent and N2 is the adsorbate with the primary measurand being specific adsorption and secondary measurands being specific pore volume, average pore diameter, frequency pore diameter and BET specific surface area .
* The new proposal will be focused on the mesoporous SiO2. Dr. Sadak presented the SiO2 materials evaluated for the KC. The selected SiO2 mesoporous solid has a type IV isotherm and showed good stability up to 800 °C based on thermogravimetric analysis.
* Measurements of specific adsorption of N2 will be determined at 9 different relative pressures. Specific surface area and specific pore volume will also be determined
* Planned timeline starts with registration of new KC by end of June/23 followed by evaluation of homogeneity and stability of sample by end of Dec./23. Call for participants commence in May/24 and samples delivered to participants by Aug./24.
* The proposed KC will be co-piloted by TUBITAK and VNIIM

Questions from participants:

* Dr. Shard raised the concern of potential participants because of the co-pilot country. For example, the UK won’t be able to participate in a KC co-piloted by VNIIM (Russia). Drs. Krumrey and Windover both noted that Germany and US won’t be able to participate either. Chairman acknowledges the difficulties in participant constraints.
* Dr. Fujimoto brings up the discussion on the frequency of maintenance. As many NMIs cannot participate in this KC. Chair enquire if there is any concerns of CMCs being greying out past the usual term limit of 5-years. Dr. Stosch enquire how likely does a CMC become greyed out? The other participants commented that it is not likely to actually have the CMCs becoming grey out even past the 5-year limit.
* Dr. Shard noted that it is generally useful to have a maintenance comparison periodically. Other participant noted that there has to be a justification to run a maintenance/repeat comparison such as to bringing in a new technique.
* The chairman enquired if the KC can be piloted by TUBITAK alone to allow most participants to able to participate in the KC. TUBITAK agreed to be the sole pilot lab.
* Chairman enquire if any institute wishes to participate. Chairman asked the SAWG contact person to bring the KC call for participants to their experts and get back to Dr. Sadak. Once all potential participants have notified TUBITAK, Dr. Sadak should notify Drs. Fujimoto and Shard.

SAWG leadership – Group photo

* Dr. Fujimoto noted that he was appointed as the SAWG chairman in 2019 and he has come to the end of this 4-year term. His term will finish at the 2023 CCQM plenary meeting. He has proposed Dr. Alex Shard to be the next SAWG chair person and he will be stepping down as the SAWG chair after his term completes.
* Chair asked for the opinion of the participants. All participants welcome Dr. Shard as the new chair of SAWG. The next chair will be appointed by the CCQM president at the CCQM plenary meeting. Dr. Fujimoto thanks all participants for their kind collaboration with him and asked for their continuing collaboration with Dr. Shard.
* Dr. Shard thanked Dr. Fujimoto for chairing SAWG during the very difficult COVID period and for his high spirit throughout his term.
* A group photo of both online and in-person participants were taken.

Direction of the SAWG

As the session focuses the discussion of the future direction of SAWG, Dr. Shard chaired the discussions in this session. Based on the discussion, Dr. Shard will develop a strategy document for the committee to review.

Dr. Windover on the CHIPs and Science Act of USA. The CHIPs and Science Act will have a budget of over $207B in the next five years. The actual dollar amount of the budget is still being decided with a small amount of funding will likely go into metrology and supporting R&D components that NIST is involved. Some funding will also go into NIST to fund instrumentation, packaging and standards work. Timeline of different actions were discussed. A list of new instrumentation has been put together from NIST and is currently awaiting funding approval. A large group of NIST staff are transferred into the CHIPS program and will impact a good fraction of the NIST program.

There can be international “friendly partners” participants in the program. Potentially up to $2B funding will be going to Metrology but not certain how much of it will be directed to the R&D. If interested in program, there is a publicly available document with specific topics listed. Among the topics listed, one is for standards and reference materials development. Another relevant publication provides a good guidance on how NIST develops reference materials and how the certificates are written. There is a new category of RGTM, which is an exploratory material, that are not yet reference materials but are good for ILC. NIST now has this guideline for materials for ILC under the RGTM (Research Grade Test Materials) category materials. At the end of the ILC, the materials can then be pushed out as reference materials. This will address the concern from the semiconductor industrial standard of using a 300 mm wafer.

Questions:

* Dr. Shard enquired about what sort of reference materials are coming down the pipeline? Dr. Windover noted there has been some calls and interests on Hf, SiGe, dopped films, high purity feedstock liquids and gases (in ppb purity quality).
* What new equipment were on the list? A: multiple XRDs, tomography tools. XRR and XRD tools capable of handling 300 mm wafer will be important to integrated into fab.
* Dr. Rossi enquired if there will be investment for people. Dr. Windover answered that yes, there will be short-term funding for hiring post-doc fellows but these are not on continuing basis.
* For the European members of SAWG, Dr. Shard wondered if there is any cross-synergy with the US Chips ACT.

Dr. Shard asked the participants if there is any thoughts on how to approach the broad scope claims. Dr. Windover noted that it is simply not sustainable to do repeated comparisons of very similar materials every 5 years. Dr. Krumrey noted that for the layer thickness determination, the CMC claim is based on the technique not specific to material. Layer thickness is well-setup within SAWG but composition is more challenging and Dr. Shard welcome the new proposal from Dr. Kim on compositional studies. As for organic materials, for example, to address food packaging to improve shelf-life. Characterization of thin-film used in food packaging with anti-microbacterial properties will be important.

SAWG currently has joint activities in the IAWG on nanoparticles, Dr. Shard asked if there is any interest in core-shell nanoparticles, in measuring the shell thickness, for the applications of display materials. Dr. Krumrey noted that PTB has some activities in this area. Jorg Radnik (BAM) has lots of interest in quantum dot materials characterization.

Dr. Shard felt encouraged by the three proposals going forward in the next few years. It would be important to examine these comparisons and how they all come together to provide enough coverage for industry and provide relevant CMCs to support the industries. Any comments to please let Dr. Shard know.

Dr. Fujimoto noted the five priorities as identified by the CCQM can be used as a broad guide. Among the topics discussed, health and life sciences haven’t been discussed too much. Dr. Fujimoto suggested a broader stakeholder engagement of both end-users and scientific community. Among the different stakeholders, there are broad engagement with those identified on the list already. SAWG has less visibility in health and safety area, US pharmacopeia, FDAs… etc. is an area that can be strengthened. Dr. Shard noted that surface functionalization of nanoparticles in Health would be of interest to SAWG community. Dr. Shard invite anyone to contact him to discuss any ideas for future pilot studies in any of the new foresighted area.

Dr. Windover noted printed standards for security reference materials (e.g. for Ion-mobility spectrometry) and measurement of a loosely bound materials on surfaces (which has some similarity to loosely bond bio-films on biological systems) and may be of interest to the SAWG community.

Dr. Fujimoto asked if there is any other business. None raised. Dr. Fujimoto recap the brief history of SAWG. He expressed his future plan and desire to continue to collaborate throughout other affiliated organization activities (such as VAMAS). Dr. Fujimoto thanked all in-person and online participants and closed the 2023 SAWG meeting.