

KRISS's activities at CCQM-SAWG

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Abstract

The high-tech industries have been developed based on science and technology, which involves precise measurements with smaller uncertainties. In particular, since even a tiny amount of dopants can change the entire properties of the semiconductor device, which industry is pursuing smaller, lighter, thinner, and shorter, accurate measurement is essential in the semiconductor industry. Recently, nanoscale thin films and nanomaterials where quantum phenomena are observed have been produced, and thus, the thickness of the thin films and the size of the nanomaterials for semiconductors will be further thinner and smaller hereafter. As the material size becomes smaller, the surface properties have an essential influence on the properties of semiconductor devices, so it is expected that the accurate measurement of surface properties at the nanoscale will become more critical in the future.

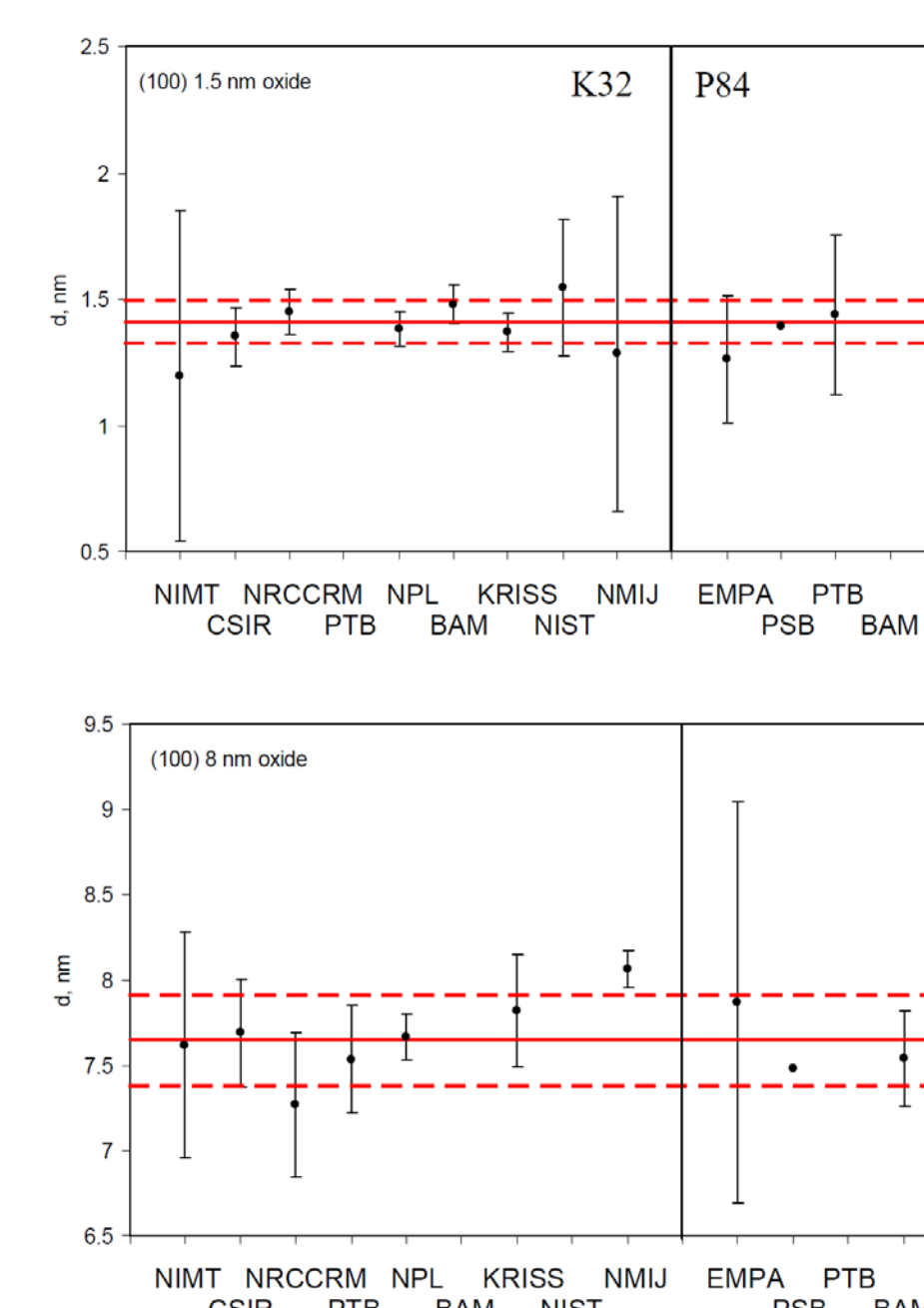
Since its launch in 2003, the Surface Analysis Working Group (SAWG) of the Consultative Committee for Amount of Substance (CCQM) has conducted seven international key comparisons (KCs). KRISS participated in the first KC of SAWG, CCQM-K32, and has played an essential role in the SAWG by conducting the past three KCs (K-67, K-129, and K-157). In addition, KRISS proposed the pilot study of 'the quantitative analysis of PtNi alloy thin films' in 2023, which was organized by nine participants from seven countries and has been successfully conducting the study so far. We hope this pilot study, P-248, to be developed as a KC in the near future. The themes of KCs conducted by KRISS have focused on the thickness measurement of thin films and the quantitative analysis of the compositions of thin films. In addition, KRISS has published papers on international key comparisons to secure measurement equality between countries, mutual comparisons between various surface analysis measurements, and correction methods discovered while performing them, such as the mutual calibration method (Metrologia 45, 507(2008)) and the uncertainty decision (Metrologia 58, 034002(2021)) in the thickness measurement of the thin film in nanoscale.

The industrial measurement standard requires quantitative analysis and measurements guaranteed in ultra-thin films, even under 10 nm. Various measurement instruments and techniques in surface analysis have been developed as the industrial demands expand to thinner thickness and smaller amounts, and comparative analysis among various measurement techniques is essential for establishing the standards in the field of surface analysis. One of the optimum instruments for measuring the properties of the ultra-thin films is the medium energy ion scattering (MEIS), which KRISS used in recent KC, K-157(Metrologia 60, 08010(2023)). As KRISS has been, KRISS aims to respond steadily to the new demand for industrial measurement standards by conducting comparative analysis in materials measurement for industry and advanced manufacturing in the future.

CCQM-K32 & P84, Amount of silicon oxide as a thickness of SiO₂ on Si

• The 1st KC of Surface Analysis Working Group

- **Proposed at the CCQM-SAWG meeting in 2004 by Dr M. P. Seah (NPL). Final report in 2008.**
- **Objective:** Evaluate the ability of National Metrology Institutes (NMIs) and Designated Institutes (DIs) to measure SiO₂ thickness on silicon wafers (1.5 nm to 8 nm)
- **Participants:** 9 participating institutes including KRISS (including P84, 13 organizations)
- **Methods:** XPS(X-ray photoelectron spectroscopy), NR(neutron reflectometry), XRR(X-ray reflectivity), and ellipsometry
- **Key Findings:**
 - Significant improvements in measurement precision.
 - Surface contamination corrections enhanced accuracy.
- **Conclusion and more:** K32 demonstrates substantial improvements in the precision and consistency of SiO₂ thickness measurements, particularly for films below 8 nm. However, surface contamination remains an important factor for absolute measurements, although the study's protocols and corrections have enhanced overall accuracy. For films thinner than 1 monolayer may be measured by XRF or SIMS with calibration using RBS.

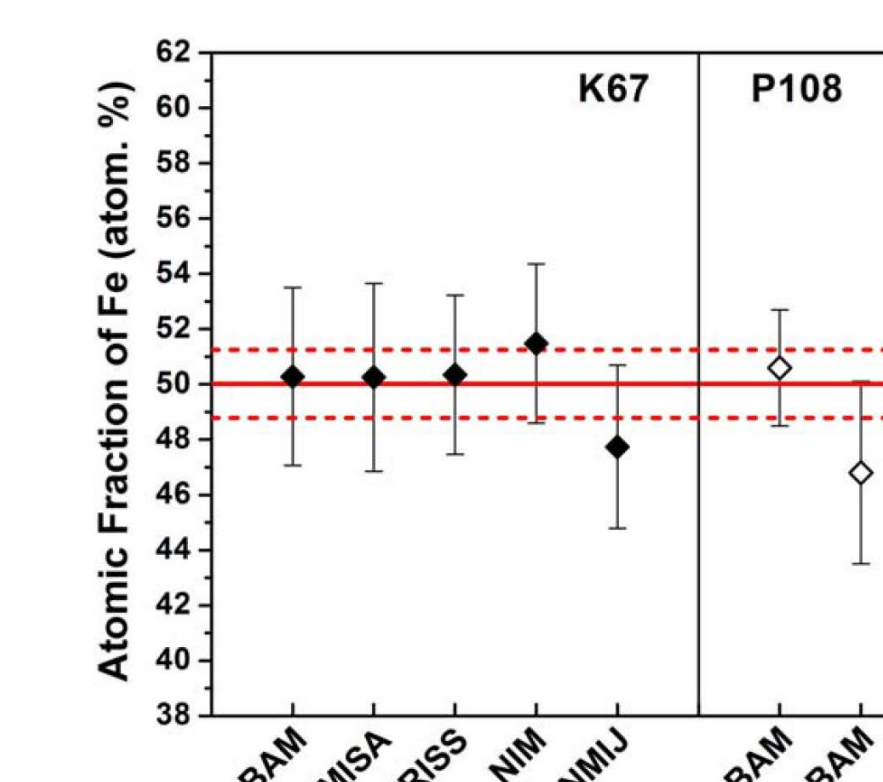


KRISS data By Dr D.W. Moon using XPS [1]

CCQM-K67 & P108, Measurement of composition of a thin Fe-Ni alloy film

• The 1st KC KRISS proposed

- **Proposed at the CCQM-SAWG meeting in 2007 by Dr K. J. Kim. Final report in 2010.**
- **Objective:** Evaluate the ability of NMIs and Dis to measure the atomic fractions of Fe and Ni in a Fe-Ni alloy film(Fe51-Ni49), expressed in atomic percent
- **Participants:** 4 NMIs and 1 DI (K67), with one DI (P108)
- **Methods:** XPS, AES, and EPMA (in P108) {ref: ICP-MS, lateral homogeneity by SIMS}
- **Key finding:** RSFs (Relative Sensitivity Factors) were determined from XPS and AES spectra, of the Fe-Ni alloy films and used to calculate the atomic composition of Fe and Ni.
- **Conclusion:** The KCRV (Key Comparison Reference Value) for the atomic fraction of Fe was calculated as 50.02% Fe with an uncertainty of 1.23%.

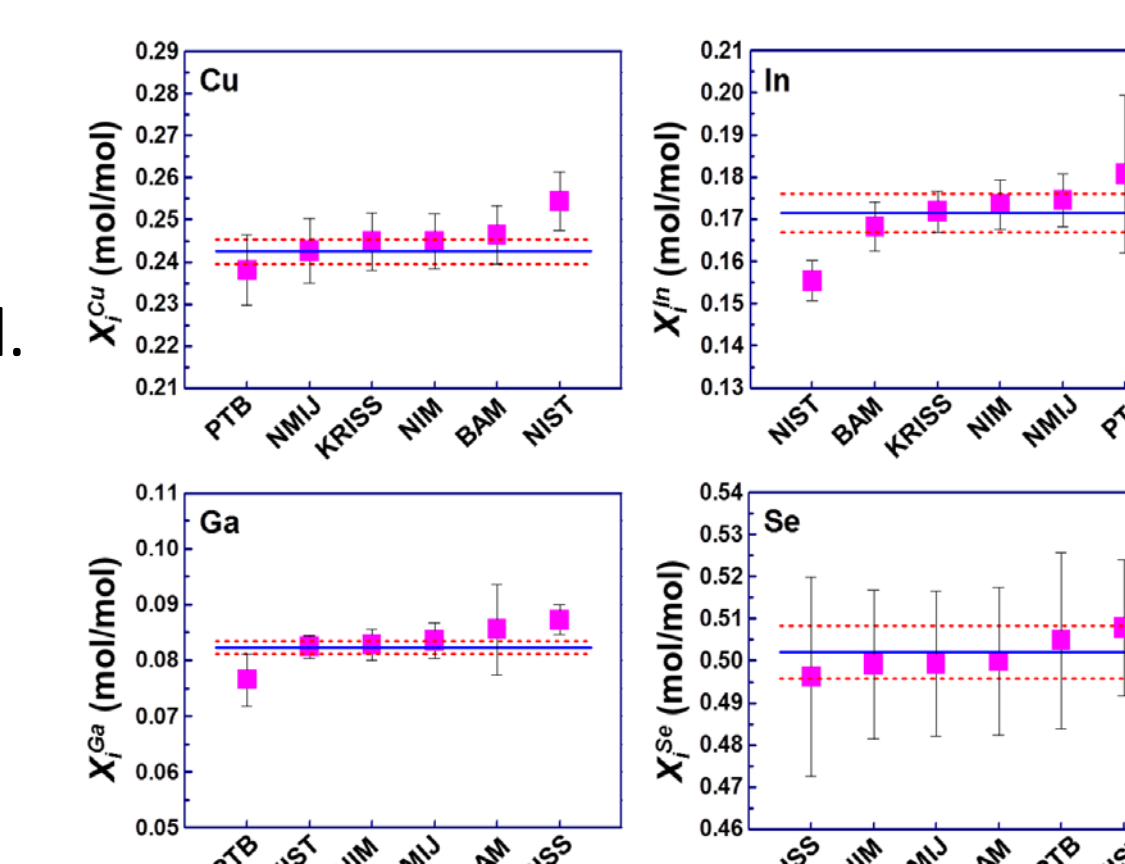


KRISS data by Drs K.J. Kim, J.W. Kim & D.W. Moon using XPS [2]

CCQM-K129 & P140, Measurement of mole fractions of Cu, In, Ga and Se in Cu(In,Ga)Se₂ Films

• Quantification of multi-element alloy film

- **Proposed at the CCQM-SAWG meeting in 2014 by Dr K. J. Kim. Final report in 2016.**
- **Objective and participants:** Evaluate the ability of NMIs and Dis to measure the mole fractions of Cu, In, Ga, and Se in Cu(In,Ga)Se₂ (CIGS) thin films. 7 NMIs & 1 DI participated.
- **Methods:** XPS, AES, SIMS, and XRF {ref: ICP-MS}
- **Key finding:**
 - The average degrees of equivalence uncertainties for Cu, In, Ga, and Se were low (ranging from 0.0047 mol/mol for Ga to 0.0228 mol/mol for Se)
 - The mole fractions measured by different laboratories were consistent, with some variation due to the methods used.
- **Conclusion:** K129 confirmed that the quantification of Cu, In, Ga, and Se in CIGS films using various surface analysis methods is feasible with high precision.

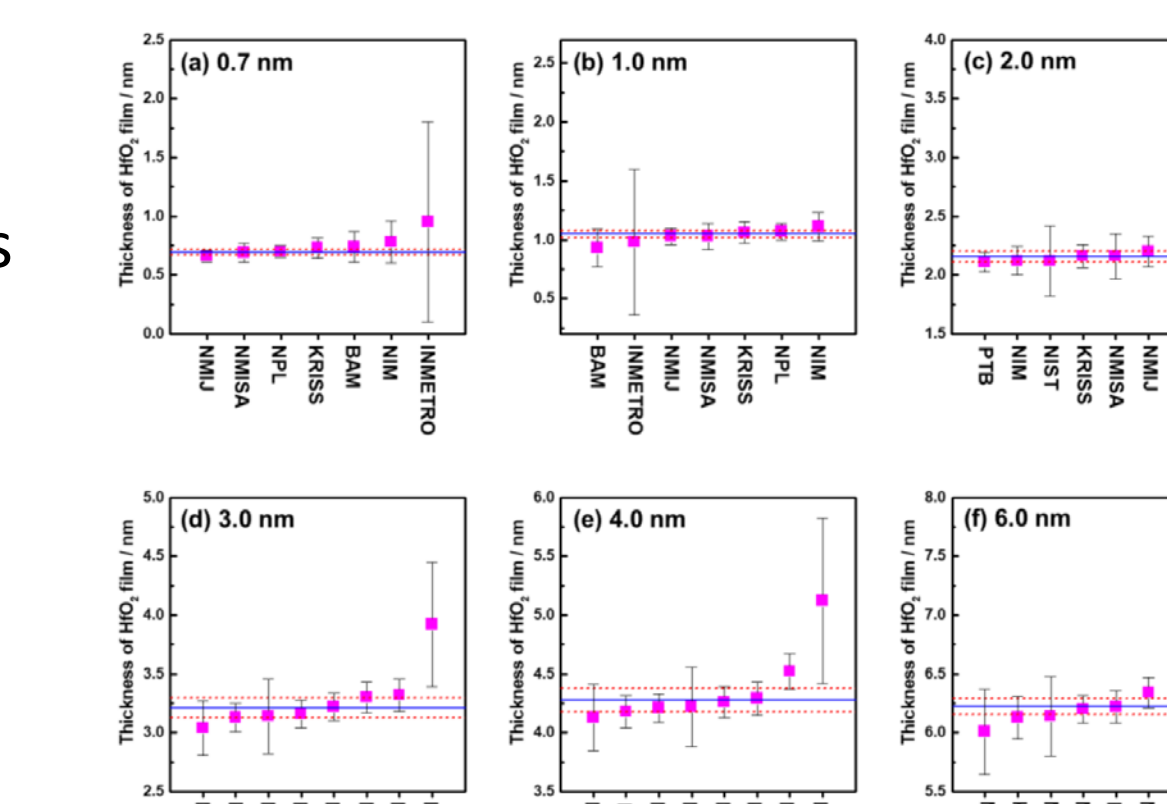


KRISS data by Drs K.J. Kim, A.S. Kim, J.S. Jang, and J.K. Suh using XPS [3]

CCQM-K157 & P190, Measurement of the amount of substance of HfO₂ expressed as the thickness of nm films

• Thickness measurement of ultra thin films

- **Proposed at the CCQM-SAWG meeting in 2017 by Dr K. J. Kim. Final report in 2023.**
- **Objective:** Evaluate the ability of NMIs and Dis measure the thicknesses of the HfO₂ films with the nominal thicknesses from 0.7 to 6.0 nm on Si (100) substrates
- **Participants:** 8 NMIs and 1 DI (K157), 10 NMIs, 1 DI, 2 others (P190)
- **Methods:** XPS, TEM, XRR, SE, AES, and MEIS
- **Key finding and conclusion:** The thicknesses of nm HfO₂ films were measured and reported by 8 NMIs and 1 DI using XPS, XRR, TEM, SE and MEIS. The expanded uncertainties of the KCRVs 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.



KRISS data by Drs K.J. Kim, H. Yu, S.M. Lee, J.H. Kwon, and H. Ruh by MEIS [4]

Conclusions

Since its launch in 2003, the Surface Analysis Working Group (SAWG) of the Consultative Committee for Amount of Substance (CCQM) has conducted seven international key comparisons (KCs). KRISS participated in the first KC of SAWG, CCQM-K32, and has played an essential role in the SAWG by conducting the past three KCs (K-67, K-129, and K-157). KRISS is currently conducting one pilot study P-248 (originally P-229.a.) about the amount of fractions of Pt in Pt-Ni alloy thin film. As KRISS has been, KRISS aims to respond steadily to the new demand for industrial measurement standards in the future.

Reference

1. "CCQM-K32 key comparison and P84 pilot study: Amount of silicon oxide as a thickness of SiO₂ on Si", M. P. Seah et al., 2008 Metrologia 45 08013. (DOI 10.1088/0026-1394/45/1A/08013)
2. "Final report on Key Comparison K67 and parallel Pilot Study P108: Measurement of composition of a thin Fe-Ni alloy film", K. J. Kim et al., 2010 Metrologia 47 08011. (DOI 10.1088/0026-1394/47/1A/08011)
3. «Final report of CCQM-K129 Measurement of Mole Fractions of Cu, In, Ga and Se in Cu(In,Ga)Se₂ Films», K. J. Kim et al., 2016 Metrologia 53 08011. (DOI 10.1088/0026-1394/53/1A/08011)
4. "Final report of CCQM-K157 for the measurement of the amount of substance of HfO₂ expressed as the thickness of nm films", K. J. Kim et al., 2023 Metrologia 60 08010 (DOI 10.1088/0026-1394/60/1A/08010)



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