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**Workshop
on
Quantitative Surface Analysis
Using XPS**

October 18 - 23, 1999

Submitted by Dr. Yongsup Park

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**Korea Research Institute of Standards and Science
(KRISS)**

1. Introduction

With the rapid increase of worldwide demand for advanced materials during the last decades, the ability to perform materials characterization has become a critical factor for the development and practical use of new and advanced materials. Particularly, quantitative elemental analysis based on electron spectroscopy techniques, such as X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) is widely used in various materials production facilities of R&D centers. The methods for quantitative analysis using these techniques, however, has become mature very recently and related activities are happening mainly in developed countries through ISO TC201 which many of the developing countries do not recognized or are ignorant of.

As part of efforts to draw attention from developing countries to these aspects of XPS quantitative analysis and eventually to attract them under ISO TC201, Korea Research Institute of Standards and Science (KRISS) proposed a project of "Workshop on Quantitative Surface Analysis using XPS" under the umbrella of the UNIDO project to establish an International Center for Materials Evaluation Technology (ICMET).

The workshop aims at providing the participants with technical knowledge of quantitative XPS analysis as well as other surface chemical analysis related to ISO 201 activities.

The workshop was organized by the Division of Chemical Metrology and Materials Evaluation, KRSS, from October 18 ~ 23, 1999.

2. Participants

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3. Instructors

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Dr. Dae Won Moon, Principal Researcher, Division of Chemical Metrology and Materials Evaluation, KRISS

Dr. Kyungjoon Kim, Senior Researcher, Division of Chemical Metrology and Materials Evaluation, KRISS

Prof. Hee-Jae Kang, Dept. of Physics, Chungbuk Nat'l Univ., Korea

4. Training Schedule (Some photographs taken during the schedule are attached in Annex A.)

Oct. 18 (Mon.)

Introduction to KRISS

Introduction to ICMET Project

Laboratory Tour (Div. of Chemical Metrology and Materials Evaluation, KRISS)

- Surface Analysis Lab.
- Crystal Evaluation Lab.
- Microstructure Science Lab.
- Materials Performance Lab.
- Epitaxial Semiconductor Lab.

Welcome Party (hosted by Dr. Yongsup Park)

Oct. 19 (Tue.)

Lectures and Practical Sessions

- Introduction to XPS (Dr. Yongsup Park)
- Certified Reference Materials (Dr. Kyung joong Kim)
- Introduction to SIMS (Dr. Dae Won Moon)
- Sputter-Depth Profiling (Dr. Kyungjoong Kim)

Oct. 20 (Wed.)

Industrial Site Tour (Samsung Electronics Co.)

Oct. 21 (Thu.)

Historical Site Tour (DMZ)

Oct. 22 (Fri.)

Lectures and Practical Sessions

- Quantitative XPS (Dr. Yongsup Park)
- Quantitative AES (Prof. Hee-Jae Kang)
- Introduction to MEIS (Dr. Dae Won Moon)
- Introduction to ISO TC 201 (Dr. Dae Won Moon)

Farewell Party (hosted by Dr. Yang-Koo Cho, Director, MEC, KRISS)

Oct. 23 (Sat.)

Lectures

- Introduction to XPS/AES related ISO Standards (Dr. Y. Park)

Discussion and Evaluation

5. Summary of Lectures

Introduction to XPS

The lecture was focused mainly on the familiarization of the participants to the techniques of XPS. The historical development of the technique was reviewed, and the instrumentation and data collection scheme needed for effective XPS analysis were explained in details. In addition, there was a lecture and practical session about useful insights to the basic ideas of XPS data analysis and interpretation through representative examples commonly encountered in practical analysis settings.

Introduction to Certified Reference Materials (CRM)

The definition of the Certified Reference Materials - for example, how they are certified and how to use CRMs to enhance the performance of various surface analysis equipment – was provided. CRMs available from KRISS and elsewhere were also introduced.

Introduction to SIMS

SIMS is a surface analysis tool most commonly used in semiconductor industries on account of its extremely high sensitivity for impurity dopant analysis. In the lecture, the basic principles of SIMS, such as sputtering processes and secondary ionization mechanism were introduced. A description was also made about the basic SIMS instruments including ion guns and mass filters, as well as typical SIMS application. At the practical session, emphasis was put on the SIMS matrix effect and sputter damage problem which makes the SIMS very difficult method for quantitative analysis. Standards used in SIMS were also reviewed with an introduction to the relevant activities of KRISS.

Quantitative XPS

Quantitative aspects of XPS analysis were lectured in details. Basic concepts were introduced on the inelastic mean free path (IMFP) and associated matrix effect corrections. Various kinds of methods for calculating and measuring IMFP were illustrated. Especially, TPP-2M method for calculating IMFP was explained in details, along with the elastic scattering correction that was recently recognized to be very important. Opportunities for hands-on calculation of matrix factor were given using Fe-Ni raw data acquired in KRISS.

Introduction to MEIS

It was explained about the development of surface/interface microanalysis of ultra-thin films with Medium Energy Ion Scattering Spectroscopy (MEIS). Growth of atomic layers on the ultra-thin films is important for the development of ULSI semiconductor devices and ultra-fast communication tools. Therefore, atomic control of ultra-thin films and subsequent surface/interface composition and structure analysis are required. MEIS was developed at KRISS to meet such demands.

The details of MEIS system of KRISS was introduced. The MEIS analysis of composition and strain distribution at gate oxides of thermal and ion beam oxides was made, and epitaxial Ge layers on Si were explained as well.

Introduction to ISO TC201

ISO TC201 was established to develop standards for surface chemical analysis. It has 8 subcommittees for terminology, general procedure, data transfer, sputter depth profiling, AES, XPS, SIMS, and GDOE. Present status and activities of TC201 were briefly explained and contribution of KRISS to ISO TC201 was described including development of reference materials for standards made in TC201 and new proposals to TC6/SIMS for estimation of SIMS depth resolution. The importance of Asian countries' participation to ISO TC201 was emphasized.

XPS/AES related ISO Standards

Various standards developed and being developed in ISO TC201 subcommittees for XPS and AES were introduced and explained.

6. Evaluation of the Workshop

For evaluation of the workshop, a questionnaire was distributed to each of the participant. The results are attached in Annex B.

7. Conclusion

The workshop, not limited to deal with quantitative XPS analysis, was intended to emphasize the importance of quantitative analysis in various surface analysis settings and encourage the participants to adopt established procedures, materials, and databases in quantitative surface analysis.

The participants showed many interests especially in the lectures on the ISO TC201 and related standards documents and responded eagerly to this topic.

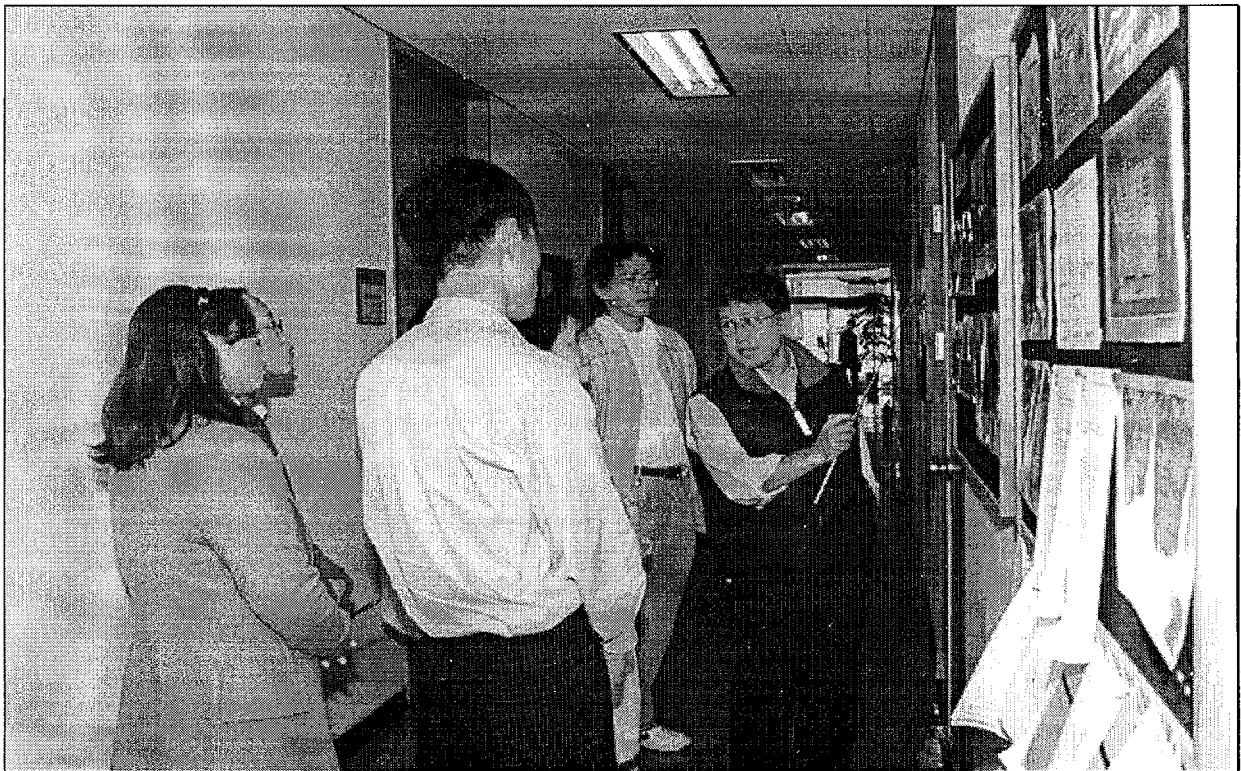
In order to facilitate and regularize this kind of workshop on materials evaluation technologies under the ICMET project, it is most important to obtain stable funds. For example, there were only a few participants with all many of the ICMET participating countries' strong interests in the workshop. This is due to the restricted funds available in their own countries.

Therefore, some portion of the expenses (for example, round trip-airfares) for the participants should be met at least by the ICMET participating institutions themselves. Any possibility of contribution from the related industries of the participating countries should be also explored for further progress of the project.

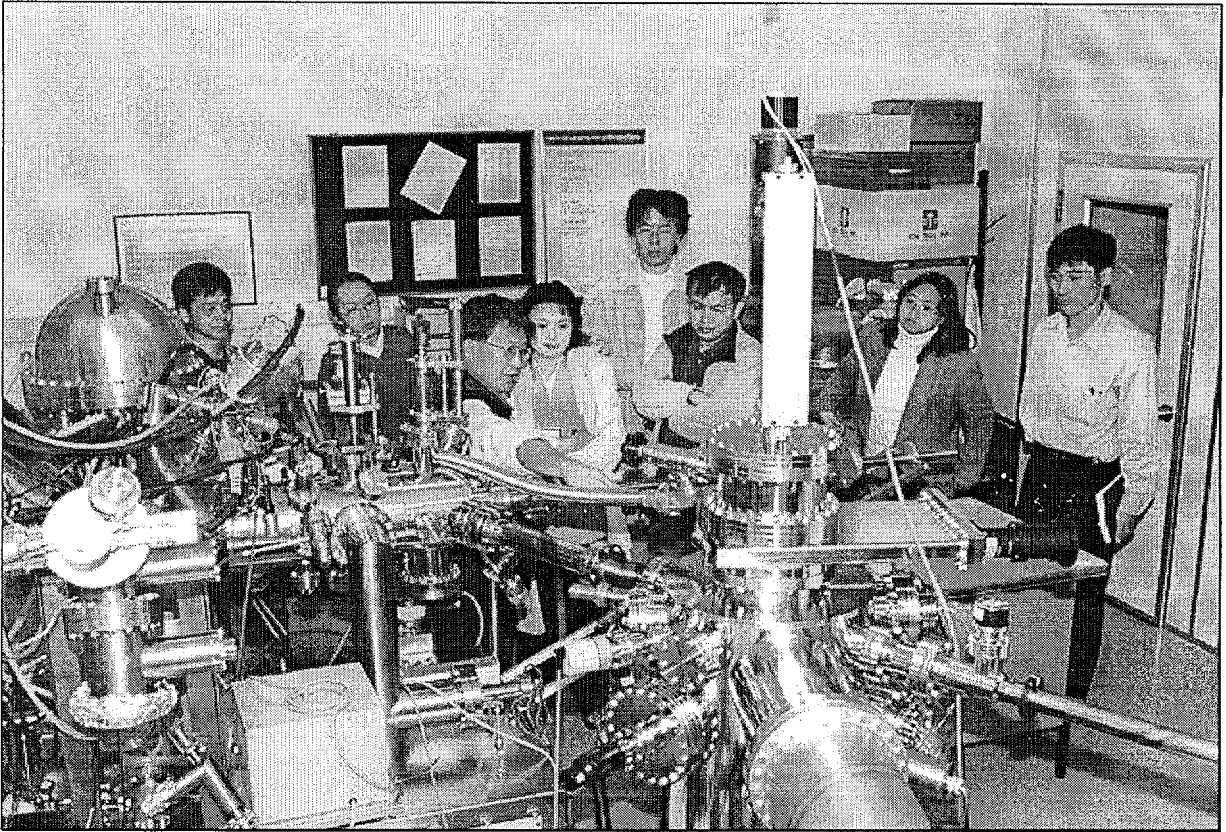
Annex A. Pictures of the workshop



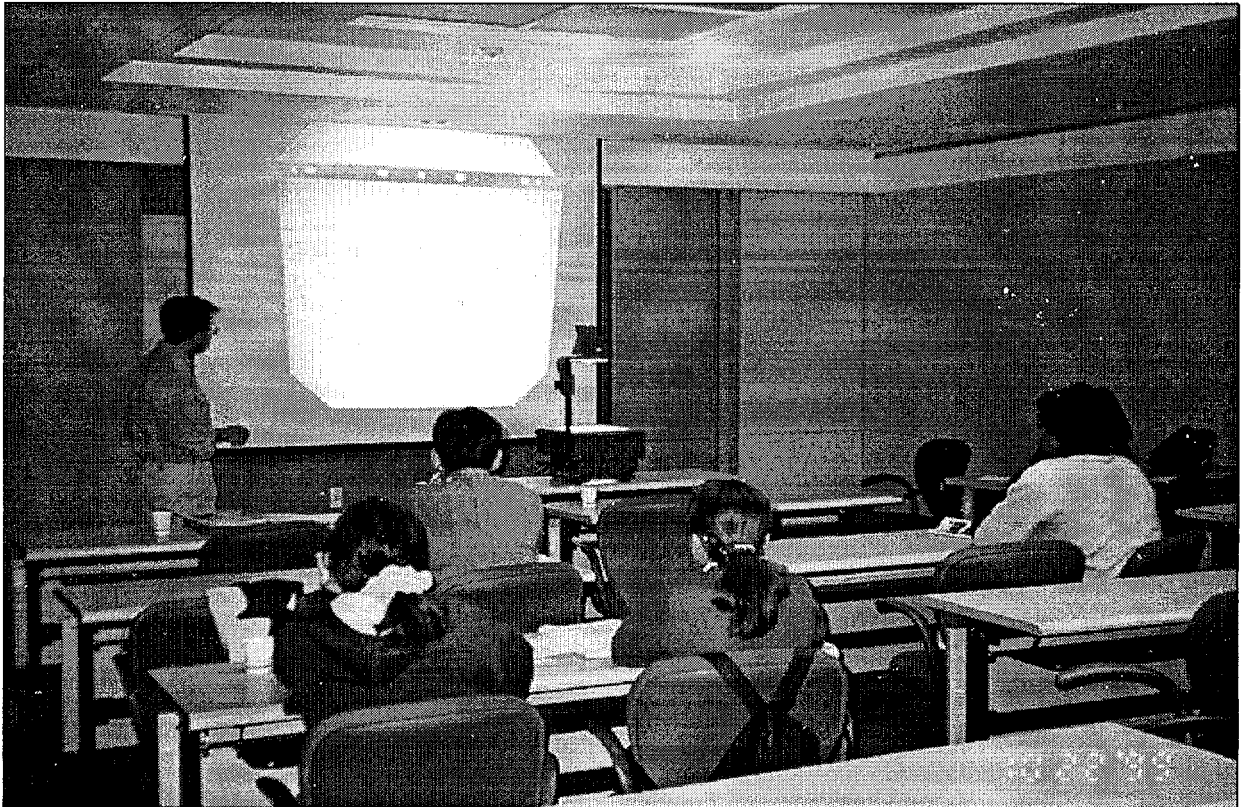
<Group picture in front of the Materials Evaluation Center>



<Laboratory tour to Surface Analysis Group>



<Practical Session about SIMS>



<Lecture about SIMS>

Annex B. Evaluation sheets answered by the participants (Attached).

Evaluation form for ICMET Workshop for Quantitative XPS

Name(print): Yu Ling

Signature: Yu Ling

1. Do you think this workshop program was helpful to you ?

Please mark below.

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(no) (yes)

Comments:

Make me clearer about ① principles of quantitative XPS.
② How to use the correction of matrix factors, sensitivity factors etc. in tested materials in order to obtain precise data.
③ the effects of depth profile with ion sputtering on chemical composition in tested samples. ④ the importance and contents of TCM documents in surface analysis.

2. How do you rate the lectures given in this workshop ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(poor) (excellent)

Comments:

(1) the contents of lectures are attractive, meaningful and wonderful.

(2)

3. Did you have any difficulty in transportation, eating, lodging, etc. during the workshop ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(yes) (no)

Comments:

Everything is very smooth.

4. Do you think ICMET project on "Quantitative Surface Analysis" should be continued ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(no) (yes)

If yes, in what subject ?

How to resolve the problems in quantitative analysis on some complicated samples, such as ^{you have met ever} insulator materials.

6. Other comments or suggestions on this ICMET workshop:

could you ~~also~~ let participants introduce their projects they have done. just make a communication each other.

Evaluation form for ICMET Workshop for Quantitative XPS

Name(print): Han-Chung Chien Signature: 簡朝中

1. Do you think this workshop program was helpful to you ?

Please mark below.

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- ⑦ ---- 8 ---- 9
(no) (yes)

Comments:

2. How do you rate the lectures given in this workshop ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- ⑦ ---- 8 ---- 9
(poor) (excellent)

Comments: Before taking the lessons, if you could provide the teaching materials, it would be helpful to us.

3. Did you have any difficulty in transportation, eating, lodging, etc. during the workshop ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(yes) (no)

Comments: Just eating. But up to now (10/23)
It's ok! We also want to thank
for the arrangement when we
arrive the Seoul intl airport.

4. Do you think ICMET project on "Quantitative Surface Analysis" should be continued ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(no) (yes)

If yes, in what subject ?

6. Other comments or suggestions on this ICMET workshop:

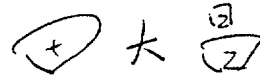
If the tours on 10/20 and 10/21 could
change to the last 2 days on the
workshop. It ~~was~~ would be perfect!

Evaluation form for ICMET Workshop for Quantitative XPS

Name(print):

Tien, Ta-Chang

Signature:



1. Do you think this workshop program was helpful to you ?

Please mark below.

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(no) (yes)

Comments:

I think that maybe add some industrial
case & application lectures is perfect!

2. How do you rate the lectures given in this workshop ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(poor) (excellent)

Comments:

It's good for "International ~~Cooperation~~ Collaboration"
, but the participants maybe too few.
Hope it will be more next time.

3. Did you have any difficulty in transportation, eating, lodging, etc. during the workshop ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(yes) (no)

Comments:

It is a good reception in ~~to~~ here,
~~to~~ nothing to complain.

4. Do you think ICMET project on "Quantitative Surface Analysis" should be continued ?

0 ---- 1 ---- 2 ---- 3 ---- 4 ---- 5 ---- 6 ---- 7 ---- 8 ---- 9
(no) (yes)

If yes, in what subject ?

Surface Analysis, Materials Analysis.
~~to~~ maybe good.

6. Other comments or suggestions on this ICMET workshop:

I suggestions that Taiwan should participate
this organization, because Taiwan (ITRI) have
many Experiments in industrial materials.
^{or}
NDL.

~~to~~