技術資料

Surface Chemical Analysis – Information Formats (Committee Draft Version)

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1 Introduction

The VAMAS-Surface Chemical Analysis Standard Data Transfer Format was approved by VAMAS Surface Chemical Analysis Community in July 1988 (Surface and Interface Analysis, 13, 63(1988)), and now this format is proposed as ISO 14976 in DIS stage. Since the importance of databases is increasing in many scientific fields, storage and manipulation of spectral data in databases become necessary. The structure of ISO 14976 is suitable for communication. but database manipulation is quite different from data communication. The additional information to ISO 14976 is necessary to handle the data in the databases, so this standard proposes three formats encoding information packages for (1) specimen information, (2) calibration information, (3) data processing information, which are important to manipulate spectral data in database. The future compatibility of the format is essential. New data format should be able to read from the old data processing systems and vice versa. Therefore, this standard is supplementary to and compatible with ISO 14976.

The objective of this paper is to notify the contents of the committee draft of "Surface chemical Analysis – Information formats" to the member of SASJ. This Version will be circulated among the member countries, and might be changed by discussions. However, we consider it is worthwhile to inform the process of the discussions.

2 General

Information is inserted into the comment lines of The VAMAS—Surface Chemical Analysis Standard Data Transfer Format(DTF) or attached to DTF as packages. As a result, the existing DTF could

be used, without alteration, as a carrier for the information packages; these packages occupy the experiment—comment line or block—comment lines in the DTF, or build blocks outside DTF.

With this structure the reading program, which utilizes those information packages, can look for the format identifiers in either the experiment—comment lines, where they apply to all blocks, or in the block—comment lines, where they apply to just one block, or outside DTF. Existing reading programs would retain these packages as text lines.

In this standard, the information packages for 'specimen information format', 'calibration information format', and 'data processing format' are described. It is module structure so that new—with—old compatibility is always maintained

2.1 Definitions and abbreviations

- (1) database: a set of retrievable spectral data
- (2) information: information about specimens and/or the procedures of analyzer calibration and/or data processing procedures and/or the information necessary to create spectral database
- (3) package: a set of text lines which describe information on spectral data
- (4) DTF: ISO NP14976: Surface chemical analysis Data transfer format
- (5) IUPAC: International Union of Pure and Applied Chemistry
 - (6) CAS: Chemical Abstracts Service
 - (7) N/A: not applicable
- (8) multiple text lines: a set of text lines which identify one item

2.2 Additional rules

The each text line is defined by ISO 14976.

3 Definitions of formats

3.1 Specimen information format

specimen information format identifier = "[ISO_Specimen_Information_Format_1995_September_22]" followed by 'carriage return';

host material = text line;

Provide a generic description of the specimen.

Text line starts with the label, "host material =".

IUPAC chemical name = text line;

Enter IUPAC chemical name of host material.

Text line starts with the label, "IUPAC chemical name=".

If the specification is impossible, enter "none", "unknown", or "N/A" following the label, "IUPAC_Chemical_name=".

chemical abstracts registry number = text line;

Enter the CAS registry number of the host material.

Text line starts with the label, "chemical abstracts registry number =".

If the specification is impossible, enter "none", "unknown", or "N/A" following the label, "chemical_abstracts registry number=".

host material composition = text line;

List the principal elements present or the chemical formula.

Text line starts with the label, "host material composition=".

When the composition expressed by weight concentrations, "wt%" is attached to 'number'. If the composition can not be specified, use "-" instead of 'number'.

bulk purity = text line;

Enter purity of material and guarantor (if possible).

Text line starts with the label, "bulk purity=".

If the specification is impossible, enter "unknown" or "N/A" following the label, "bulk purity = ".

known impurities = text line;

List impurity name(s), concentration(s), and guarantor (if possible).

Text line starts with the label, "known_impurities=".

If the specification is impossible, enter "none", "unknown" or "N/A" following the label, "known_impurities=".

structure = text line:

Include information such as a description of the crystal lattice and orientation, e.g. hexagonal close-packed, and/or comments such as fracture surface at grain boundary, etc.

Text line starts with the label, "structure = ".

If the specification is impossible, enter "unknown" or "N/A" following the label, "structure = ".

form of products = text line;

Give a form of products that the specimen is used for.

Text line starts with the label, "form of products=".

If the specification is impossible, enter "unknown" or "N/A" following the label, " $form\ of\ products=$ ".

supplier = text line;

Provide the name of the manufacturer and/or supplier of the host material or give a reference to how the host was made.

Text line starts with the label, "supplier=".

If the specification is impossible, enter "unknown" or "N/A" following the label, "supplier = ".

lot number = text line:

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Provide the code that identifies the production run, etc.
                   Text line starts with the label, "lot number=".
                   If the specification is impossible, enter "unknown" or "N/A" following the label, "lot_number=".
         homogeneity = text line;
                   Select one of the following items. When supplemental comment is necessary, the character, ";",
is inserted between the selected item and the attached comment
                   homogeneity=homogeneous - homogeneous
                   homogeneity = inhomogeneous - inhomogeneous
                   homogeneity=unknown
                                             unknown
                   homogeneity = N/A
                                             - not applicable
                   homogeneity = (other)
                                              - propose other specification
         crystallinity = text line;
                   Select one of the following items. When supplemental comment is necessary, the character, ";",
is inserted between the selected item and the attached comment
                   crystallinity=single
                                           - single crystal, together with orientation (connected by " ")
                   crystallinity=poly
                                          - polycrystalline
                   crystallinity=amorphous - amorphous
                   crystallinity=unknown - unknown
                   crystallinity = N/A
                                          - not applicable
                   crystallinity=(other)
                                           - propose other specification
         material family = text line;
                   Select one of the following items. When supplemental comment is necessary, the character, ","
is inserted between the selected item and the attached comment.
                   material_family=metal
                                                   metal
                   material family=inorganic
                                                   - inorganic compound
                   material family=organic
                                                   - organic compound
                   material family=polymer
                                                    polymer
                   material_family=semi
                                                    - semiconductor
                   material family=bio
                                                    - biological material
                   material family=composite
                                                   - composite
                   material family=super_conductive - super conductive material
                   material family=(other)
                                                   - (propose a new family)
         special material classes = text line;
                   Select one of the following items. When supplemental comment is necessary, the character, "".
is inserted between the selected item and the attached comment
                   special material classes=rod
                                                      - rod or ingot
                   special material classes=sheet

    sheet or foil (without substrate)

                   special material_classes = film_single - single layer thin film or coating (on substrate),
                   special_material_classes=film_multi - multi layered thin film or multi layered coating
                                                      (on substrate)
                   special material classes=sinter
                                                      - sintered material
                  special material classes=wafer
                                                      - wafer
                  special_material_classes=powder
                                                     - powder
                  special material classes = fiber
                                                     - fiber
                  special material classes = (other)
                                                      - (propose a new class)
         specimen mounting = text line;
                   Select one of the following items. When supplemental comment is necessary, the character, ",",
is inserted between the selected item and the attached comment.
                  specimen mounting=mechanical
                                                             - mechanically mounted using screw, spring or etc.
                  specimen mounting=mechanically under grid - mechanically pressed to a grid by a spring
                  specimen_mounting=conductive adhesive
                                                              - fixed by conductive adhesive material
                  specimen mounting=nonconductive adhesive - fixed by non-conducting adhesive material
                  specimen mounting=powder compact In
                                                             - powder compact in indium foil, indium pressure pad
                  specimen_mounting = powder_put_into
                                                              - powder put into a conductive material
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(ex: hole in copper block)
specimen_mounting=(other) - (specify other method)
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ex situ preparation = text line;

Select one of the following items. If ex situ preparation is carried out by the series of the following items, multiple text lines are adopted, and the label for each text line are numbered as "ex_situ_preparation_1=" or "ex_situ_preparation_2=". The number indicates the order of ex situ preparation. If different kinds of ex situ preparations are carried out simultaneously, ex situ preparation procedures are combined with "+" following the single label, "ex_situ_preparation=". When supplemental comment is necessary, the character, ";", is inserted between the selected item and the attached comment.

```
ex_situ_preparation=none - none
ex_situ_preparation=polish - polish
ex_situ_preparation=cleavage - cleavage
ex_situ_preparation=ion - cut by ion beam
ex_situ_preparation=powder_compact_steel_pad - powder_compact_using_steel_pressure_pad
ex_situ_preparation=acetone - degreased_by_acetone
ex_situ_preparation=(other) - (specify_other_method)
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in situ preparation = text line;

Select one of the following items. If in situ preparation is carried out by the series of the following items, multiple text lines are adopted, and the label for each text line are numbered as "in_situ_preparation_1=" or "in_situ_preparation_2=". The number indicates the order of in situ preparation. If different kinds of in situ preparations are carried out simultaneously, in situ preparation procedures are combined with "+" following the single label, "in_situ_preparation=". When supplemental comment is necessary, the character, ";", is inserted between the selected item and the attached comment.

charge control condition = text line;

Select one of the following items. If charge control procedures are carried out by the series of the following items, multiple text lines are adopted, and the label for each text line is numbered as "charge_control_condition_I=" or "charge_control_condition_2=". The number indicates the order of charge control procedures. If different kinds of charge control procedures are carried out simultaneously, each charge control condition is combined with "+" following the single label, "charge_control_condition=". When supplemental comment is necessary, the character,";" , is inserted between the selected item and the attached comment.

specimen temperature = text line;

Enter ambient temperature or heating temperature. (not necessary to consider the temperature change by a primary beam flux). The temperature is expressed by the unit "K", and "K" is attached with 'number'.

Text line starts with the label, "specimen_temperature = ".

If the specification is impossible, enter "specimen_temperature=unknown".

comment on specimen information = text line;

Text line starts with the label, "comment =". If multiple text lines are necessary, each text line starts with the numbered labels as "comment I =" or "comment 2 =".

If there is no comment, it is not necessary to attach any word after "comment=".

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end of specimen information format identifier = "[end_of_specimen_information_format]" followed by 'carriage return';
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3.2 Calibration information format

calibration information format identifier = "[ISO_Calibration_Information_Format_1995_September_22]" followed by 'carriage return';

energy scale calibration = text line;

Indicate the technique, element name with transition used for the calibration. Text line starts with the label, "energy scale calibration feature label=". The technique and element name are connected with "_". Following this text line, indicate energy scale label and peak value. Text line starts with the label, "energy scale calibration feature nominal energy=". The energy scale label and peak value are connected with "_". If calibration is done by using multiple peaks, each technique and element name are indicated with the numbered label as "energy scale calibration feature label 1=", and energy scale label and peak value are indicated with the numbered label, "energy scale_calibration feature_nominal_energy_1 = ". This combination is followed by "energy scale calibration feature label 2=" and "energy scale calibration feature nominal energy 2=", and so on.

When the charge control condition is carried out, indicate the referencing element name with transition and referencing peak value following the text line label, "energy_scale_calibration_charge_compensation=". The referencing element name and peak value are connected with "_". When flood gun is used, enter "flood" instead of referencing element name.

When a spectrum is not calibrated, enter "energy scale calibration=uncalibrated".

intensity scale calibration = text line;

Specify the intensity scale calibration procedure. It is acceptable only to indicate the name of referred name of journal(s) or document(s).

Text line starts with the label, "intensity_scale_calibration=". When multiple text lines are necessary to describe the procedure, the numbered labels are used as "intensity_scale_calibration_l=" or "intensity scale calibration 2=".

When a spectrum is not calibrated, enter "intensity scale calibration=uncalibrated".

resolution calibration = text line;

Specify the resolution scale calibration procedure.

Text line starts with the label, "resolution_calibration=". When multiple text lines are necessary to describe the procedure, the numbered labels are used as "resolution_calibration_1=" or "resolution_calibration_2=".

When a spectrum is not calibrated, enter "resolution calibration=uncalibrated".

end of calibration information format identifier = "[end_of_calibration_information_format]" followed by 'carriage return';

3.3 Data processing information format

data processing information format identifier =

"[ISO_Data_Processing_Information_Format_1995_September_22]"

followed by 'carriage return';

data processing procedure = text line;

Specify the data processing procedure.

Text line starts with the label, "data_processing_procedure ="

When different data processing procedures are carried out sequentially, each data processing procedure is indicated in one text line with numbered labels as "data_processing_procedure_l=" or "data_processing_procedure_l=". The number indicates the order of the data processing.

When a spectrum is not processed, enter "data_processing_procedure=unprocessed".

end of data processing information format identifier = "[end_of_data_processing_information_format]" followed by 'carriage return';

Annex A(informative)

Annotation

A.1 host material

Provide a generic description of the specimen, such as stainless steel, gold copper alloy, 6061 Al, polyamide, nylon, alumina, or gallium arsenide. For layered structures, the host material is the "bulk" substance near the surface. For instance, XPS of an ultra—thin metal film on a thick SiO2 layer on a Si substrate would be 'silica' because the XPS would not probe the Si. The examples are listed in Surface Science Spectra, 1, 141(1992).

A.2 host material composition

List the principal elements present or the chemical formula. If the composition can not be specified, use "-" instead of 'number', and when it is expressed by weight concentrations, "wt%" is attached to 'number', e.g. Li-P-O-, Li3PO4, SiO2, W(CO)6, or Fe74Cr18Ni8wt%. Examples are listed in Surface Science Spectra, 1, 141(1992).

A.3 bulk purity

Enter purity of host material and the name of guarantor(if possible) and the unit is attached with the number, e.g. 99.99wt% by NISSAN ARC LTD. The units acceptable are wt% and atomic%. The expression like "4N" is not preferable.

A.4 known impurities

List impurity name(s), concentration(s) and the name of guarantor(if possible), and the unit(s) are attached with the number(s), e.g. N:0.01wt%, O:0.02wt% checked by NISSAN ARC LTD., or S:4E17atoms/cm3. The units acceptable are as follows; wt%, atomic%, ppm, ppb, atoms/cm3, and atoms/cm2.

A.5 form of products

Give a form of products that the specimen is used for, e.g. MOSFET, reagent, magnetic disk, single-crystal wafer, stub from corroded fender, lubricant film on the hard disk etc.

A.6 energy scale calibration

When the XPS energy calibration is carried out using Cu, Ag, and Au, then the expression is as follows;

"energy_scale_calibration_feature_label 1=XPS_Cu2p3/2"

"energy_scale_calibration_feature_nominal_energy_1=BE_932.66eV"

"energy_scale_calibration_feature_label_2=XPS_Ag3d5/2"

"energy_scale_calibration_feature_nominal_energy_2=BE_368.27eV"

"energy_scale_calibration feature label 3=XPS Au4f7/2"

"energy_scale_calibration_feature_nominal_energy_3=BE_84.00eV"

If energy calibration is carried out by the charge compensation procedure by referencing C1s peak value, expression is as follows;

"energy_scale_calibration_charge_compensation=C1s 285eV"

In the case of AES, the example is as follows;

"energy_scale_calibration feature label 1=AES CuMVV"

"energy_scale_calibration_feature_nominal_energy_1=KE_61.16eV"

"energy_scale_calibration_feature_label_2=AES_AuNVV"

"energy_scale_calibration_feature_nominal_energy_2=KE_72.21eV"

"energy_scale_calibration_feature_label_3=AES_CuLVV"

"energy_scale_calibration_feature_nominal_energy_3=KE_918.62eV"

The peak values for the energy scale calibration for XPS and AES are listed in Surf. Interface Anal., 14, 488(1989), and Surf. Interface Anal., 15, 293(1990), respectively.

A.7 intensity scale calibration

Specify the intensity scale calibration procedure. It is necessary to indicate the name of referenced name of journal(s)

or document(s). Intensity scale calibration procedures are reported in J. Surf. Sci. Soc. Jpn., 15, 376(1994), J. Surf. Sci. Soc. Jpn., 16, 434(1995), and J. Electron Spectrosc. Relat., 50, 137(1990), or NPL A1; NPL calibration procedure for AES, or NPL X1; NPL calibration procedure for XPS.

A.8 energy resolution calibration

Specify energy resolution scale calibration procedure. It is recommended to use simple expressions. "FWHM of Ag3d5/2:0.97eV" means that "The energy resolution of the electron energy analyzer is estimated by the full width at the half maximum of silver peak(Ag3d5/2) which equals 0.97eV."

A.9 data-processing procedure

Specify data—processing procedure. It is acceptable to use abbreviations like 'S-G' instead of 'Savitzky-Golay', or 'Tougaard' instead of 'Tougaard background subtraction'. One processing procedure is written in one text line.

Annex B(informative)

Examples

B.1

[ISO Specimen Information Format 1995_September_22] host material=polyethylene IUPAC chemical name=polyethylene chemical_abstracts_registry_number=9002-88-4 host material composition = C2H4 bulk_purity=99.5wt% checked by NISSAN ARC LTD. known impurities=0:0.3wt%, N:0.1wt% checked by NISSAN ARC LTD. structure=none form of products=supermarket bag supplier=Mitsubishi Chemical Co. lot number=961017PE homogeneity = homogeneous crystallinity = amorphous material_family=polymer special_material_classes=sheet specimen mounting=mechanically under grid ex_situ_preparation=degreased by n- hexane in_situ_preparation=none charge_control_conditions = flood+screen specimen temperature = 298K comment=sample is linear low density polyethylene sheet [end of specimen information format] [ISO_Calibration_Information_Format_1995_September_22] energy scale calibration feature label 1=XPS_Cu2p3/2 energy scale calibration feature nominal_energy_1=BE_932.7eV energy scale calibration feature label 2=XPS Au4f7/2 energy_scale_calibration_feature_nominal_energy_2=BE_84.0eV energy_scale_calibration_charge_compensation=flood_6eV intensity scale calibration=NPL X1 resolution_calibration=FWHM of Ag3d5/2:0.97eV [end_of_calibration_information_format] [ISO Data Processing Information Format 1995 September 22] data_processing_procedure_1=smoothing by 5 points Savitzky-Golay data_processing_procedure_2=Shirley background subtraction [end of data processing information format]

B.2

[ISO_Specimen_Information_Format_1995_September_22]
host_material=indium_gallium_arsenide
IUPAC_chemical_name=N/A
chemical_abstracts_registry_number=none
host_material_composition=In0.52Ga0.48As
bulk_purity=99.999wt% checked by NISSAN ARC LTD.
known_impurities=S:1.8E17 atms/cm3 checked by NISSAN ARC LTD.
structure=cubic; a=0.5868nm
form_of_products=laser diode
supplier=Japan Energy
lot_number=#2845
homogeneity=homogeneous
crystallinity=single_(100)
material_family=semi
special_material_classes=film_multi; total_thickness = 50nm

specimen mounting=mechanical; with 4 screws ex_situ preparation=ethanol in_situ_preparation=ion 2kV_10uA Ar charge control conditions=none specimen temperature=298K comment=atomically flat interface [end_of specimen information format] [ISO Calibration Information Format 1995 September 22] energy_scale_calibration_feature_label_1=XPS_Au4f7/2 energy scale calibration feature nominal energy 1=BE 84.00eV energy scale calibration feature label 2=XPS Cu2p3/2 energy_scale calibration feature nominal energy 2=BE 932.67eV intensity scale calibration = uncalibrated; refer to Cu and Au wide range spectra acquired together resolution_calibration=FWHM of Ag3d5/2:0.78eV [end_of_calibration_information_format] [ISO Data Processing Information Format 1995 September 22] data_processing_procedure=subtraction of X-ray ghosts [end_of_data_ processing information_format]

B.3

[ISO_Specimen Information Format 1995 September 22] host material=strontium chloride IUPAC chemical name=strontium dichloride chemical_abstracts_registry_number=0476-85-4 host material composition=SrC!2 bulk purity=99.9wt% checked by NISSAN ARC LTD. known_impurities=N:0.01wt%, O:0.02wt% checked by NISSAN ARC LTD. structure=cubic fluoride; a=0.698nm form of products=unknown supplier=Johnson Matthey lot number=EP01007 homogeneity = homogeneous crystallinity=poly material family=inorganic special material classes=powder specimen mounting=powder compact In ex situ preparation=none in_situ_preparation=ion_2kV_10uA_Ar charge control conditions=none specimen temperature = 298K comment= [end of specimen_information format] [ISO Calibration Information Format 1995 September 22 energy scale calibration feature label 1=AES CuMVV energy scale calibration feature nominal energy 1=KE 61.16eV energy_scale_calibration_feature_label_2=AES_AuNVV energy scale calibration feature nominal energy 2=KE 72.21eV energy scale calibration feature label 3=AES CuLVV energy scale calibration feature nominal energy 3=KE 918.62eV intensity scale calibration=J. Surf. Sci. Soc. Jpn., 15, 376(1994) resolution_calibration=uncalibrated [end of calibration information format] [ISO Data Processing Information Format 1995 September 22] data processing procedure 1=smoothing by 7 points Savitzky-Golay data_processing_procedure_2=Shirley background subtraction [end of data processing_information_format]

B.4

[ISO Specimen Information Format 1995 September 22] host material=stainless steel IUPAC chemical name=unknown chemical abstracts registry_number=unknown host material composition=Fe74Cr18Ni8wt% bulk purity=99.9wt% checked by NISSAN ARC LTD. known_impurities=N:0.01wt%,O:0.02wt% checked by NISSAN ARC LTD. structure=face centered cubic; a=0.359nm form of products=sink supplier=Johnson Matthey lot number=No 15876 purchased on 18 May 1993 homogeneity = homogeneous crystallinity=poly material_family=metal special material classes=sheet specimen_mounting=mechanical ex situ preparation 1=polish ex situ preparation 2=acetone in_situ_preparation=ion_2kV_10uA_Ar charge_control_conditions=none specimen temperature=298K comment=corroded [end_of_specimen_information_format] [ISO Calibration Information Format 1995_September_22] energy scale calibration feature label 1=AES CuMVV energy scale calibration feature nominal energy 1=KE 61.16eV energy scale calibration feature label 2=AES CuLVV energy scale calibration feature nominal energy 2=KE 918.62eV intensity_scale_calibration=J. Surf. Sci. Soc. Jpn., 15, 376(1995) resolution calibration=uncalibrated [end_of calibration information format] [ISO Data Processing Information Format 1995 September 22] data_processing procedure = smoothing by 7 points Savitzky-Golay [end_of_data_ processing information format]

B.5

[ISO_Specimen_Information Format 1995_September_22] host_material=carbon overlayer IUPAC chemical name=none chemical_abstracts_registry_number=none host material composition=C bulk_purity=99.99wt%, same as target; hot isothermal pressed carbon known_impurities=O, N, F structure=amorphous form_of_products=magnetic disk supplier=DENKI KAGAKU KOGYO KABUSHIKI KAISHA lot_number = DA2150-AC04, 15 Oct.1996 homogeneity = homogeneous crystallinity=amorphous material_family=inorganic special_material_classes=film_single specimen mounting=mechanical ex situ preparation 1=stamping out ex_situ_preparation_2=acetone in_situ_preparation=ion 2kV 5nA Ar; ion sputtered for surface cleaning charge control conditions=none specimen_temperature=298K

comment 1=diamond-like protective carbon layer comment_2=magnetic disk having lubricating layer [end_of_specimen_information_format] [ISO_Calibration_Information_Format_1995_September_22] energy_scale_calibration_feature_label_1=XPS_Cu2p3/2 energy_scale_calibration_feature_nominal_energy_1=BE_932.66eV energy_scale_calibration_feature_label_2=XPS_Ag3d5/2 energy_scale_calibration_feature_nominal_energy_2=BE_368.27eV energy_scale_calibration_feature_label_3=XPS_Au4f7/2 energy scale calibration feature nominal energy_3=BE_84.00eV intensity_scale_calibration=J. Surf. Sci. Soc. Jpn., 16, 434(1995) resolution_calibration=uncalibrated [end_of_calibration information format] [ISO_Data_Processing Information_Format_1995_September_22] data_processing_procedure_1=smoothing by 5 points S-G data_processing_procedure_2=Tougaard Background Removal(B=2866eV2, C=1633eV2) [end_of_data_processing information_format]

Bibliography

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- (4) M.P.Seah, G.C.Smith, and M.T.Anthony; Surf. Interface Anal., 15, 293(1990)
- (5) M.Yoshitake and K.Yoshihara; J. Surf. Sci. Soc. Jpn., 15, 376(1994)
- (6) M.Yoshitake and K.Yoshihara; J. Surf. Sci. Soc. Jpn., 16, 434(1995)
- (7) M.P.Seah; J. Electron Spectrosc. Relat., 50, 137(1990)