
Surface Analysis Forum Newsletter No. 25

Autumn 1998

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National Physical Laboratories
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AGM of The UK Surface Analysis Forum

Acknowledgements

Announcement of the forthcoming July 1999 Meeting of the Surface Analysis Forum

***"Data Processing and Data Systems:
What Real Progress has been made since the early 90's?"***

7th July 1999

Host: Dr. Albert Carley, Chemistry Dept., University of Wales (Cardiff)

The User Group meeting at Cardiff will focus on recent advances in data analysis in the field of surface analysis. A meeting was held in Cardiff in the early 1990's on this topic, but given the advances in computer systems in the last decade, the committee feel it is time to examine whether any true progress has been made. The papers should be approximately 20 minutes in duration, certainly no longer. The committee would be particularly interested in papers submitted by postgraduate students, although as stated above, all submissions will be considered on their individual merits.

The committee is also interested in possible workshop titles for the Cardiff meeting. These do not have to be run by the proposer of the title, but a suggestion of a possible chairman of the workshop would be gratefully received.

Please NOTE! The papers are requested for the **July 1999 meeting, NOT the January 1999 meeting at Shell**

Please forward a title and a brief abstract **before the 20th of December 1998** to:-

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"The Characterisation of Thin Layers and Depth Profiles by Angle Resolved XPS"

National Physical Laboratories - Wednesday 15th July 1998

The NPL meeting was unique in the history of the UK Surface Analysis Forum, in that the afternoon session involved a hands on workshop on the use of the NPL spreadsheet developed by Dr Peter Cumpson. The unusual nature of the meeting resulted in a larger than usual number of delegates attending the meeting.



Figure 1. Delegates to the UK Surface Analysis Forum summer meeting at NPL



Figure 2. Bob Wild presenting Dr. M. P. Seah with a prize to mark the release of the first ISO standard in Surface Analysis, "The Data Transfer File Format"

The meeting started promptly at 10 am, with a short presentation by Dr Lea on the work of the NPL group. Dr Wild then stopped the meeting to present Dr Martin Seah (NPL) with a prize from the group, to acknowledge his outstanding efforts in developing the first ISO standard in the field of Surface Analysis, the Data Transfer Format.

"Composition Depth Profile Data by ARXPS"

Dr P Cumpson, NPL

Dr Peter Cumpson (NPL) presented the first Paper on the use of ARXPS, its application and its limitations. Dr Cumpson, defined the research programme on ARXPS at NPL and charted the development of the spreadsheet programme which



Figure 3. Dr. Peter Cumpson (NPL) giving the first paper at the UK Surface Analysis Forum Meeting at NPL

was evaluated in the afternoon workshop.

Peter went through the advantages and disadvantages of ARXPS measurements, namely that for most instruments it is possible to perform the experiment, but difficult to do the subsequent analysis. Over the 20 years in which the technique has been used, 20 different models have been developed in order to construct surface profiles. Most of these models only really work on selected systems. Peter has tried to take the best models and insert them into a user-friendly interface in order to make the technique more accessible to non-experts. Dr Cumpson then

showed some basic examples, and demonstrated how the software would handle these types of system. From their studies, NPL believe that they have developed a basic “toolkit” for the surface analyst which will make the data obtained from ARXPS measurements, more accurate and reliable.

Some Industrial Analytical Case Studies: A Menu of Approaches Including Angle-Resolved Methods

Dr G Smith, Shell

Dr Smith described the industrial application of ARXPS and the method of analysis currently adopted by his Research group at Shell, Thornton Laboratories. Dr Smith initially described the basic overlayer model developed by Fadely in 1984 (*CS Fadely, Progr.Surf.Sci., 16(1984),3*), and pointed out some of the basic limitations in this approach, specifically what value of λ to use? Where:

$$D_A(\theta) = \lambda_A \cos\theta \cdot \ln[1 + (I_A \cdot I_B) / (I_B \cdot I_A)]$$

For a two layer model system

At Shell they use the attenuation depth rather than the Inelastic Mean Free Path (IMFP), as defined in the Seah and Cumpson paper (SIA 1997). Dr Smith showed some basic examples initially SiO_x on Si and subsequently the absorption of lubricant layers on iron oxide (i.e. Lubricant/FeO_x/Fe System). This particular system which is of significant importance to Shell, has a background problem making the analysis more difficult than usual. Most commercial packages would fit a Shirley background to the data, but using the QUASES software it was possible to fit a Tougaard background to the data. The different backgrounds gave different lubricant film thicknesses, of 3.74nm (Shirley) and 3.9nm (Tougaard), the latter corresponding to the values of film thickness determined using other methods of analysis.



Figure 4. Dr. Graham Smith from Shell

For further information on the data given in Dr Smith's talk please see the original overheads (860k) or read the relevant sections in "Quantitative Surface Analysis for Materials Science", GC Smith, Institute of Materials, 1991. ISBN 0-901462-79-9.

Application of QUASES for the non-destructive depth profiling of implanted SiO₂ films in Si

Prof. M Prutton, University of York



Figure 5 Prof M Prutton presenting his ARAES paper at the NPL Meeting.

Professor Prutton showed the analysis of a specially developed reference sample generated by Professor Mark Dowsett's group at Warwick University, using ion beam implantation. Using an SiO₂ reference sample and the QUASES software, they were able to predict the film thickness using the OKLL Auger line and compare the results with measurements made using EDX, plus the thickness determined using implantation data supplied by Professor Dowsett. The results were all in good agreement, but the data analysis using the QUASES software took 4 hours, which was considerably longer than the acquisition time!

Curve Fitting in Angle Resolved XPS using Pre-Determined Shirley Backgrounds

Prof. JE Castle, Surrey University

The work looked at the intrinsic tail in the XPS spectrum, which is normally ignored by standard commercial packages since they tend to use a pre-determined Shirley background. The group at Surrey University have looked at this addition to the background in the spectrum, for a wide number of different elements as thin overlayers and linked the tail effect to their Tougaard background subtraction method. Professor Castle showed the analysis of Ag thin films using the Surrey University peak-resolving package, which uses Voigt functions. A real life example was then studied, namely the presence of a passive film on the surface of a stainless steel. Using the Surrey peak fitting software, an accurate assessment of the Fe2p peaks could be made, even given the steeply rising background. The entire Fe2p region could be realistically accounted for in the peak fitting model, which is not possible with commercial packages. A further advantage of the software package is that only a 20 eV window either side of the main lines is required for the Tougaard background subtraction routine, most packages require a spectral window of ~100eV away from the main lines.



Figure 6 Prof. Castle at the start of his presentation on Curve Fitting.

The Analysis of layered structures by Angle resolved XPS: Modelling the data

Dr. L Hazell, CSMA

Len Hazell went through the standard method of data analysis done by most analysts. He then pointed out



Figure 7. Dr Len Hazell at the beginning of his battle with the NPL visual "aids" facilities

that nearly all of us “assume” that the surface is homogeneous within the sample volume analysed, however, we all know that in most cases this just is not true. So why do we do it? The answer is that it is just too difficult to construct a more accurate description of the surface even if we have the data. In order to describe the composition of the surface in more detail, we need to vary either q or l (see Dr Smith’s paper above), and in general we alter q , if the sample substrate is flat. Dr Hazell then described the interactive modeling programme developed by BP/CSMA, which is used at CSMA to analyse ARXPS data. The software is interactive and allows

the rapid assessment of the data, which is inputted via an EXCEL spreadsheet. The model does require some prior knowledge of the likely structure, including estimates of the possible stoichiometry of the layers and their density. The most important input parameters included:

- Common Sense
- Prior Knowledge
- Patience
- Inspiration, &
- Porsperation!



The Manufacturers Latest Innovations making ARXPS a Practicable Method of Analysis

VG Presentation (*Dr K Robinson*)

VG have recently launched the Σ_{Probe} , which can be used to perform ARXPS measurements simply by altering the input lens voltages. This means that no stage/sample movement is required and that no refocusing of the X-ray spot is needed. Also the charge compensation only has to be done once, not for each take-off angle as with older monochromator systems.



PHI Presentation (*Dr Uli Rolli*)

Dr Rolli showed the basic geometry of the Quantum 2000 spectrometer. The instrument uses a fully compensated automatic stage, which can be programmed to do ARXPS measurements, with the sample always focussed in the X-ray spot. The collection angle is set via a mechanical aperture on the analyser input lens.



Kratos Presentation (*Dr C Blomfield*)

The latest magnetic lens systems produced by Kratos, has an in-built charge neutraliser which produces a constant flux of electrons focussed on the sample surface. No adjustment is needed when the sample take-off angle is altered since the system automatically compensates. Also running the instrument in the electrostatic mode, the instrument can adjust its input lens voltages automatically and collect ARXPS data without moving the specimen.

Workshops

XPS Datasystems: "What we analysts really need is ..."

Workshops run by:-

Dr A Carley (*University of Wales (Cardiff)*)

&

Dr J Day (*Bristol University*)

Leader : *AF Carley*

This workshop focussed on the acquisition and processing aspects of datasystems, particularly in the context of quantification and precision. Although data acquisition is an apparently straightforward aspect of a datasystem which should have been sorted out by now, in fact this topic occupied most of the discussion. Turnaround times can be minimised by means of the Harrison-Hazell acquisition strategy (1992) which estimates the optimum number of scans required for a particular precision, but no commercial datasystem incorporates it. Other suggestions were for real-time trial quantification of survey scans, interleaved scanning and more flexible interaction with the process during acquisition. The ISO Standard Data Transfer Format, implemented on all commercial datasystems, meant that users were not limited to one system for processing, and indeed a show of hands indicated that most users did not confine themselves to a single system for the complete acquisition to quantification sequence. Statistical errors arising from intensity measurement and curve-fitting, which can be estimated using published algorithms, were generally ignored, and were apparently not demanded by customers. Systematic errors in the quantification process were much more difficult to address. A consensus developed in the workshop that the only way to make manufacturers aware of the failings of their datasystems, and take our suggestions seriously, was for the UK Surface Analysis Forum to produce a synthesis of the member's views and submit this to the manufacturers. Members will be canvassed for their suggestions in due course.



Figure 8 Dr Carley hard at work in the first workshop.

Leaders : *John Day and Keith Hallam*

The intention of this workshop was to identify the needs of users and to build on the experience of previous systems to identify future requirements. The workshops were introduced by describing what currently existed and addressed the hardware, data acquisition, manipulation, quantification and presentation. There was some discussion

regarding the platform used, from PDP11 to PC but the main complaint here was that manufacturers had changed their platforms and their software but not necessarily improved it. Data acquisition must be able to acquire spectra, depth profile, angle resolve and map. The hope was expressed that acquisition could become more intelligent. Within data manipulation, the various forms of smoothing, background subtraction and peak synthesis were discussed. Within presentation and storage it was felt that software systems could more easily export data to spreadsheets and word processing packages and that interchange of data had still some way to go. The adoption of the VAMAS data transfer format recognised the need for this but work must now start on producing a better transfer format. Quantification was also discussed. Most people now prefer to quantify using absolute data from fitted peaks. It was concluded that there was still a long way to go before we have an ideal data system.



Hands-on computer-based ARXPS workshop

Workshop run by Dr P Cumpson



Figure 9 Several "Senior" surface analysts who are discovering that they know less than they thought they did!



Figure 10 Even experienced users benefited from the new software!

The practical workshops were very well attended and were a major success. The software was shown to be very robust and easy to use, although this varied depending upon the package used. The general view was that the product was excellent and when will it be available to the members of the user group?



Annual General Meeting of The UK Surface Analysis Forum

Dr RK Wild thanked the members for attending the meeting, and thanked all the speakers in the last year for their excellent presentations. He noted that numbers attending the meeting were increasing, with the last two meetings having over 100 delegates in both cases. This was a record for the group and justified the slight change in structure of the meetings, where practical workshops were included.

Dr Kathy England the UK ESCA User groups treasurer then gave a brief report on the financial position of the group, which from the current balance sheet was shown to be very good. Both of the last two meetings should have made substantial profits, which enabled the group to keep the meeting fees to a minimum.

The secretary's report was given by Dr Steven Harris and praised the continuing efforts of Dr Simon Morton in taking the standard report and converting the document into a glamorous Web page. He also pointed out that the official membership list had now risen

to ~390, of which ~65 were retired members. The last two meetings had shown a dramatic increase in new younger members who were essential if the group is to continue to flourish in the year 2000 and beyond!

Elections

The following were elected to the main committee:-



Figure 11. Steve Evans thanking the Group for awarding him the John Riviere Prize at the Bristol UK ESCA User Group meeting

- Dr. RK Wild, Chairman
- Dr. SJ Harris, Secretary
- Dr. K England, Treasurer

Dr Wild announced at the end of the meeting that this would be the last time that he would stand as Chairman and that a new Chairman would be appointed in two years time. The final part of the meeting, was a brief thank you from Dr Steven Evans who was awarded the John Riviere Prize at the Bristol Meeting, he concluded his speech by asking the Chairman if he would be kind enough to sign the certificate this time!

Acknowledgements

The committee would like to thank Dr Alan Carrick, of Acolyte Science for supplying the photographs of the delegates to the NPL meeting. However, the committee are not offering a prize to the first member who correctly identifies the two distinguished members who insisted on being in most of the shots!

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