

- Atomic Layer Deposition in Long Capillaries,
- Counting Silanols on Fused Silica,
- Evaluating the XPS Peak Fitting and
Accompanying Reporting in the Literature,
and
- Uniqueness Plots in XPS

Matthew R. Linford

Department of Chemistry and Biochemistry

Brigham Young University

Provo, UT 84602

Outline

Five Projects

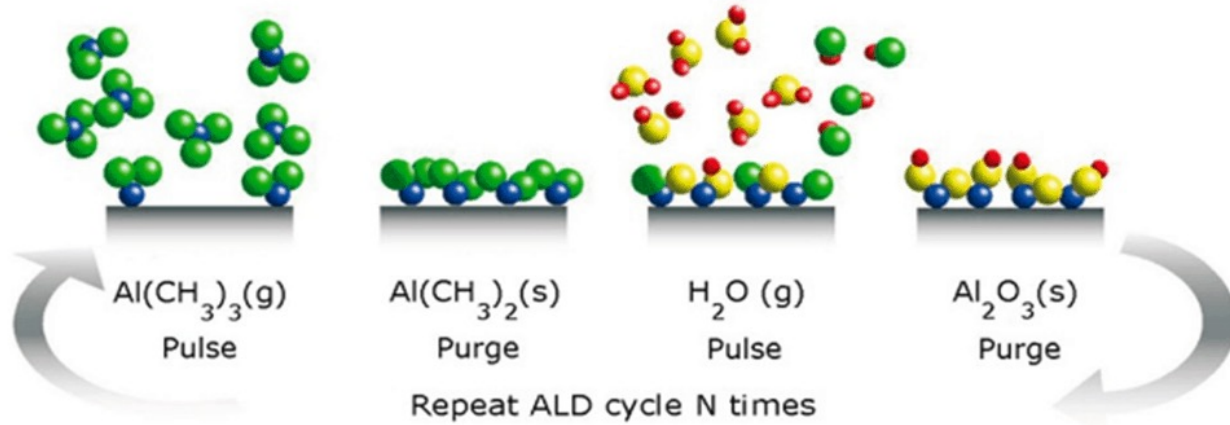
1. Atomic layer deposition in long capillaries
2. Quantifying the number of silanols on fused silica surfaces
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4. Uniqueness plots in XPS
5. Lack of experimental reporting in XPS

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What is Atomic Layer Deposition (ALD)?



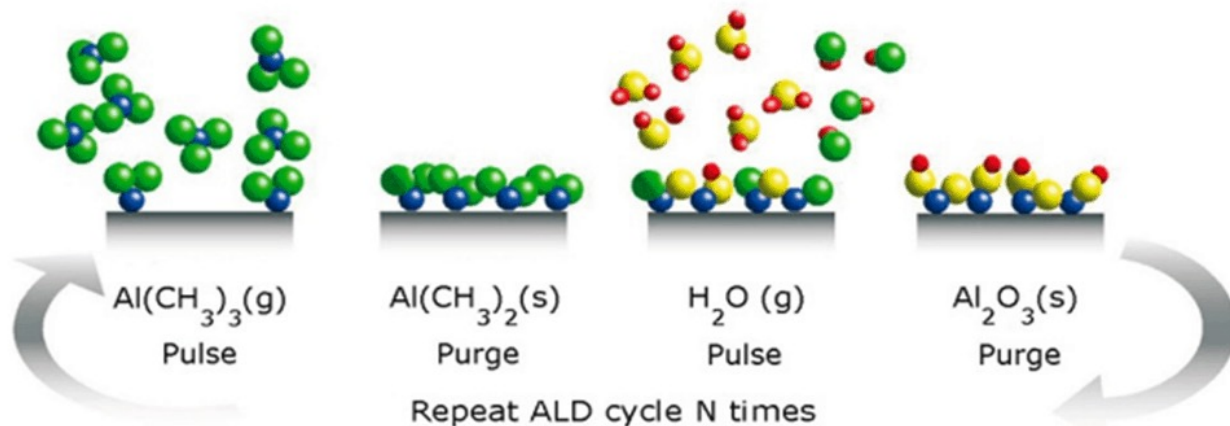
Ideally, ALD:

- Gives reproducible results
- Is based on self-terminating half-reactions
- Shows limited side reactions and secondary reactions, e.g., parasitic CVD



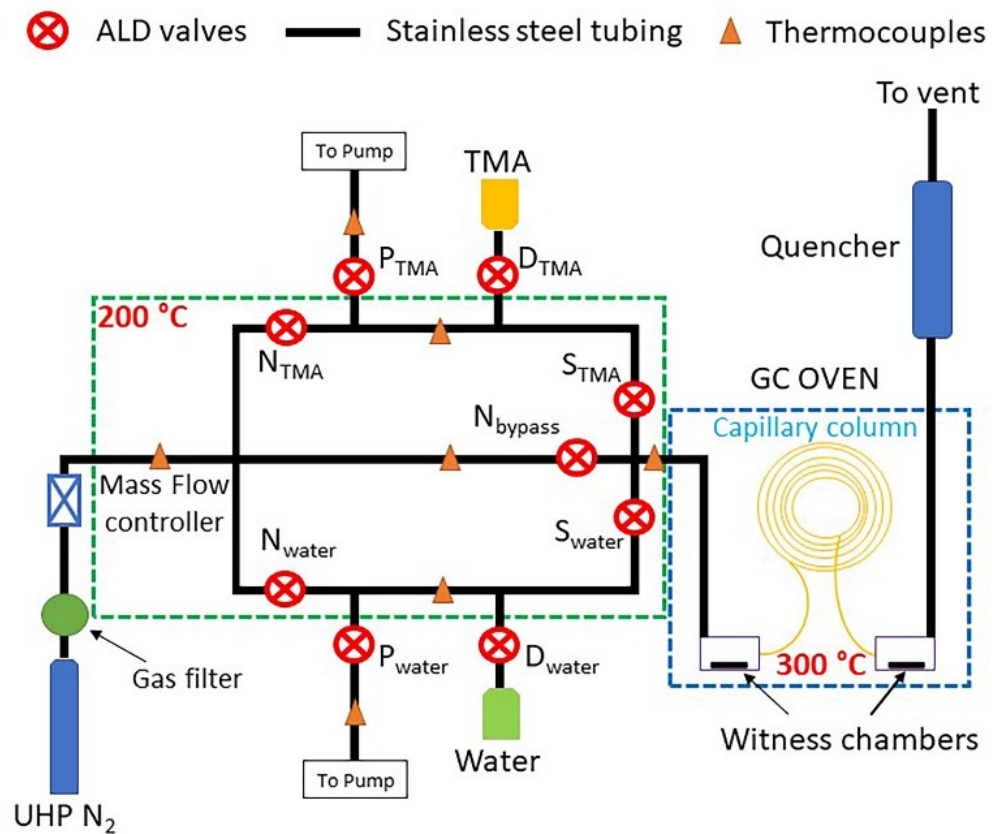
ALD in Long Capillaries

- Wanted to coat insides of 5 and 12 m capillaries (0.53 mm inner diameter)
 - High aspect ratio
 - Can we use conventional ALD?



RESTEK
Pure Chromatography

AP-ALD in Long Capillaries



UI Figure

Individual Controllers Operation

Cycles: 50

TMA Pump Time (s): 4

TMA Dose (s): 0.02

Purge Through TMA Chamber (s): 45

Secondary Nitrogen Purge (s): 15

Water Pump Time (s): 4

Water Dose (s): 0.015

Purge Through Water Chamber (s): 90

Secondary Nitrogen Purge (s): 60

Final Purge Time: 300

Control Panel

Nitrogen Bypass: ☒ On ☐ Off

☒ Safety Check

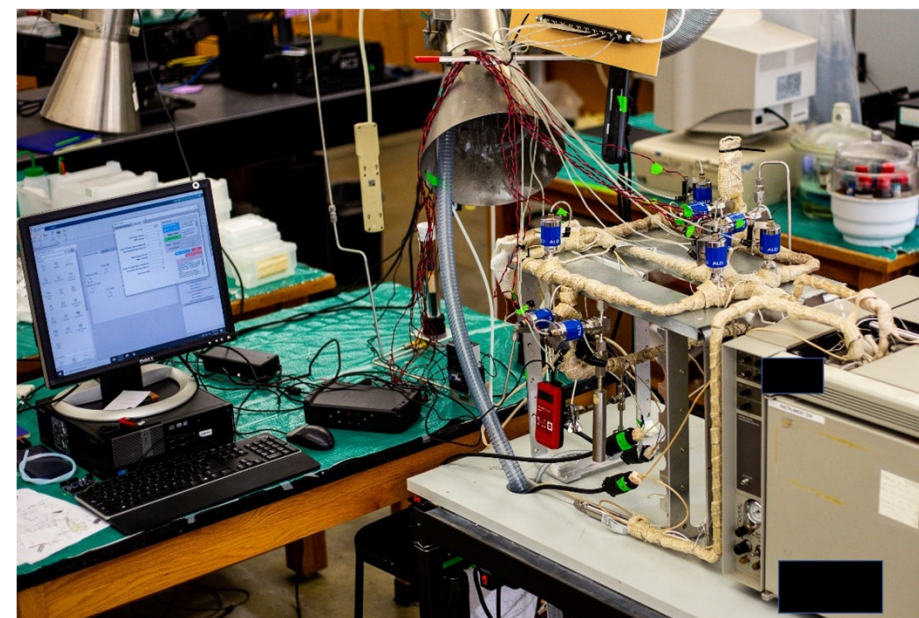
Run Program

Status: Nitrogen Valves Open

Current Cycle: 0

Abort Final Purge Bypass

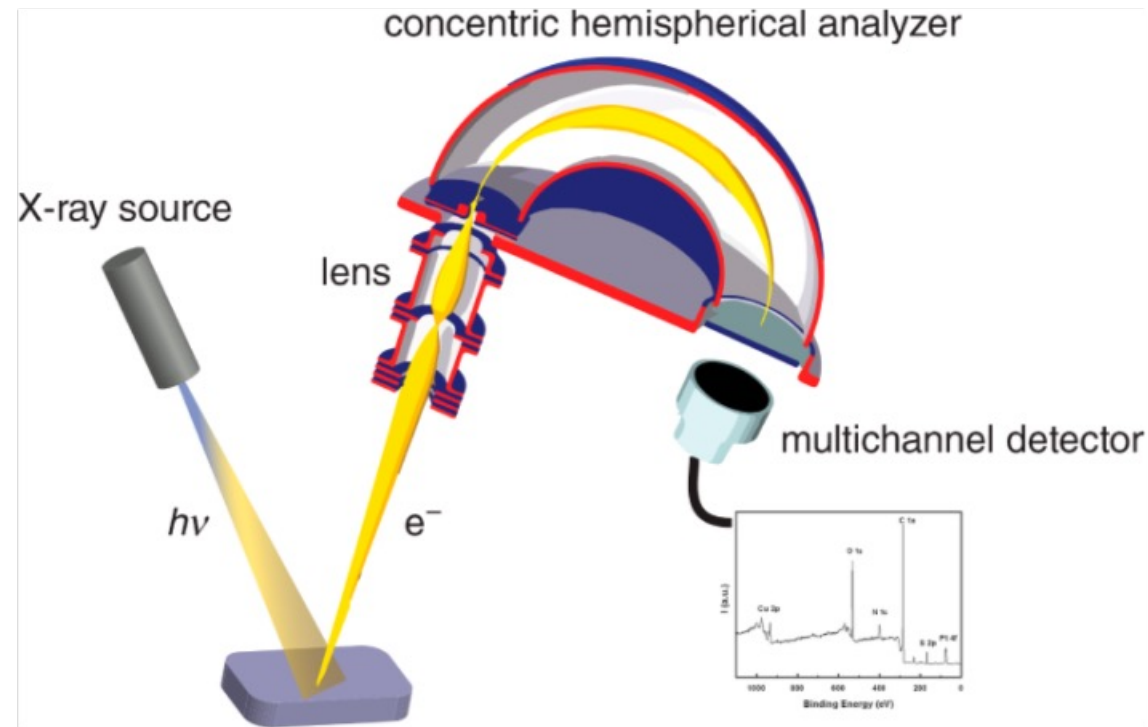
Pressing abort will not immediately stop the run, but will terminate after a purge (between TMA and water, or water and TMA). Final Purge Bypass will run based on the amount of time in the Final Purge Time Window.



Patel, Major, Jacobsen, Shah, Strohmeier, Shollenberger, Bell, Argyle, Linford. "Flow Through Atmospheric Pressure – Atomic Layer Deposition (AP-ALD) Reactor for Thin Film Deposition in Capillary Columns." *Anal. Chem.* **2022**, 94, 7483–7491.

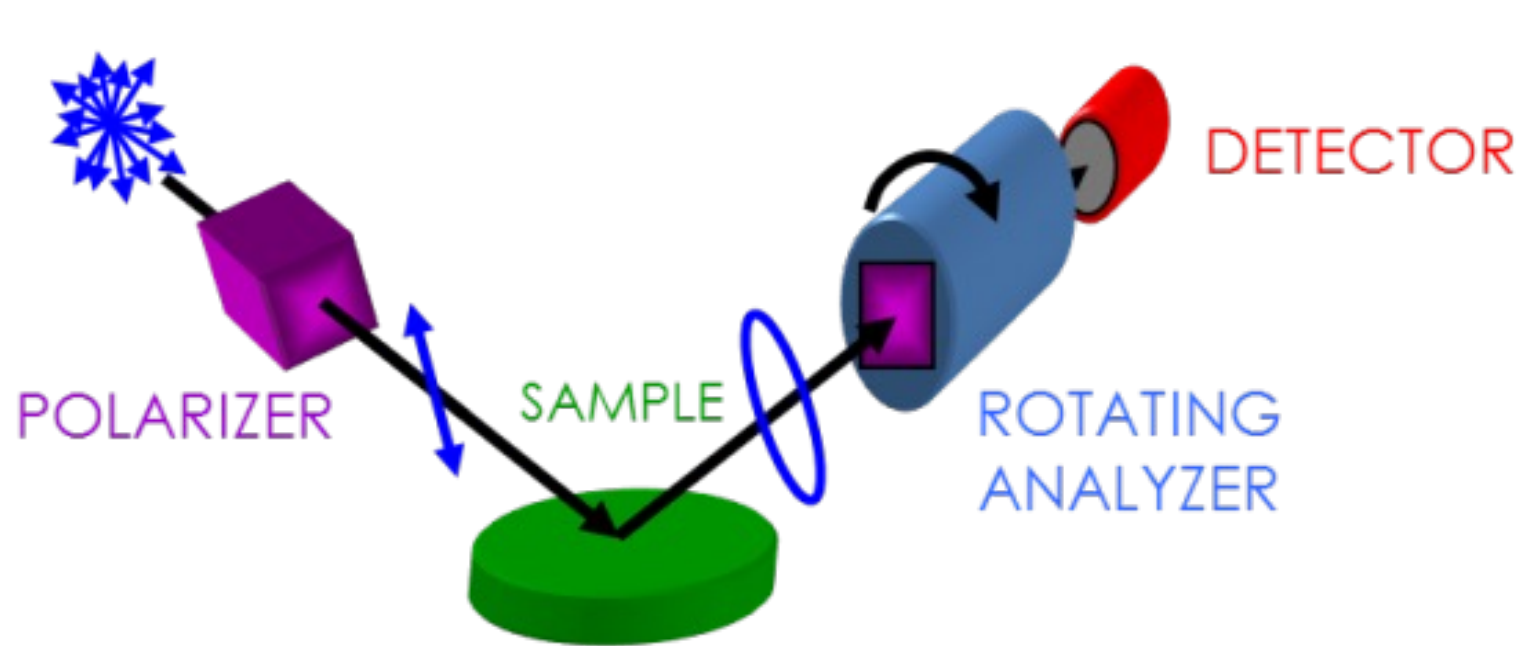
X-ray Photoelectron Spectroscopy (XPS)

- Sensitive to outermost 5 – 10 nm of samples
- Quantitative
- Detects all elements except H and He
- Provides chemical state information
- Imaging possible
- Depth profiling possible

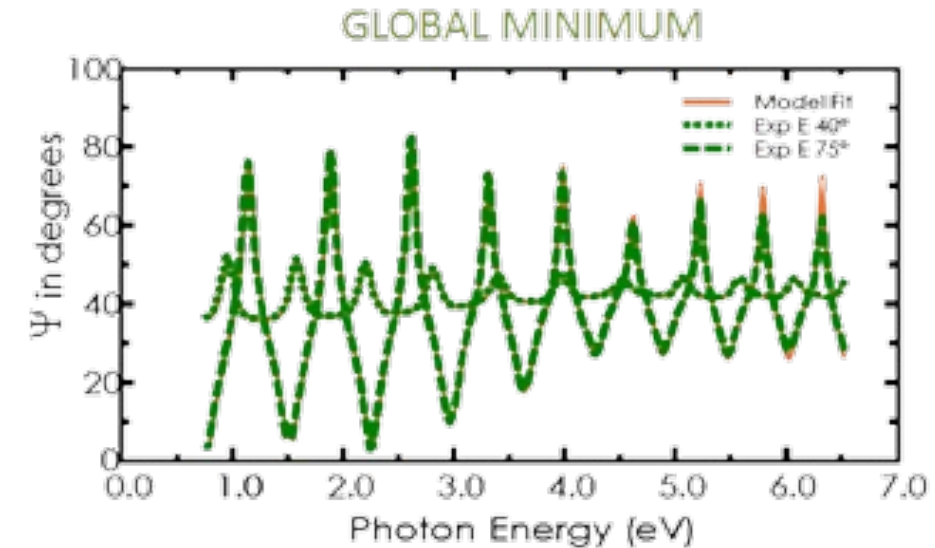


$$h\nu = BE_F + \Phi_{spec} + KE_{spec}$$

Spectroscopic ellipsometry (SE)

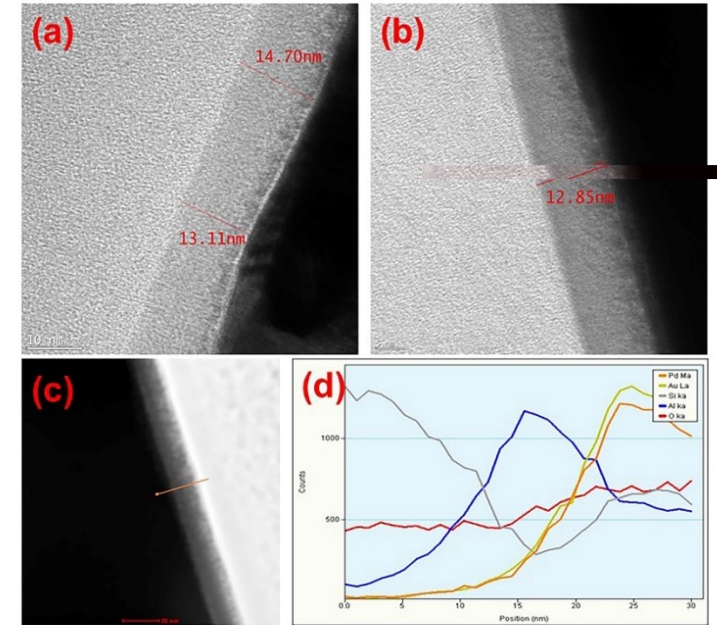
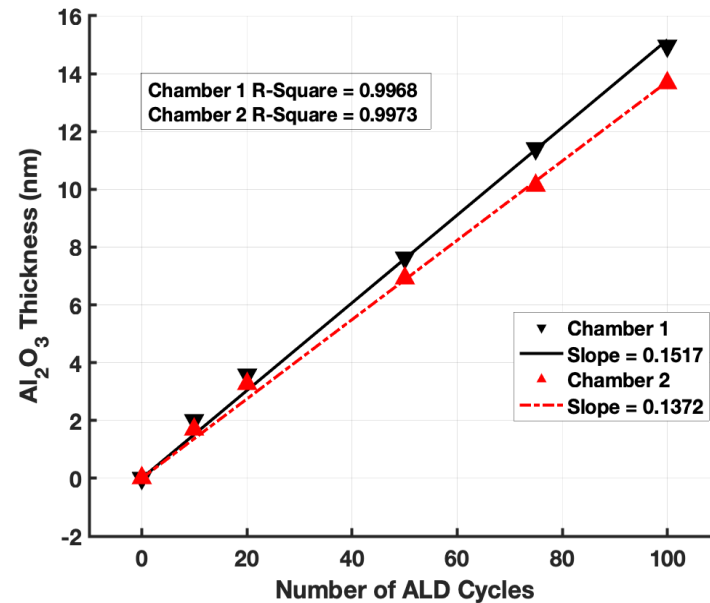
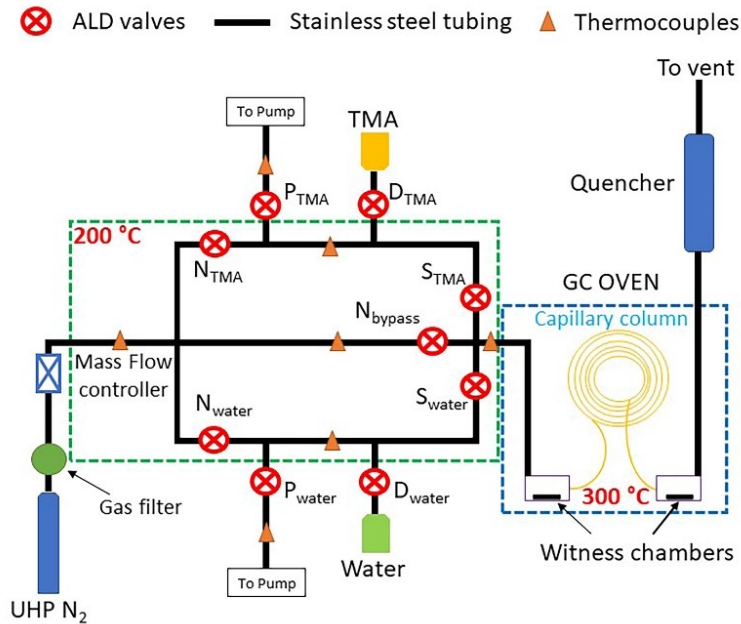


$$\rho = \tan(\Psi)e^{i\Delta}$$



SE has an 'inverse' problem

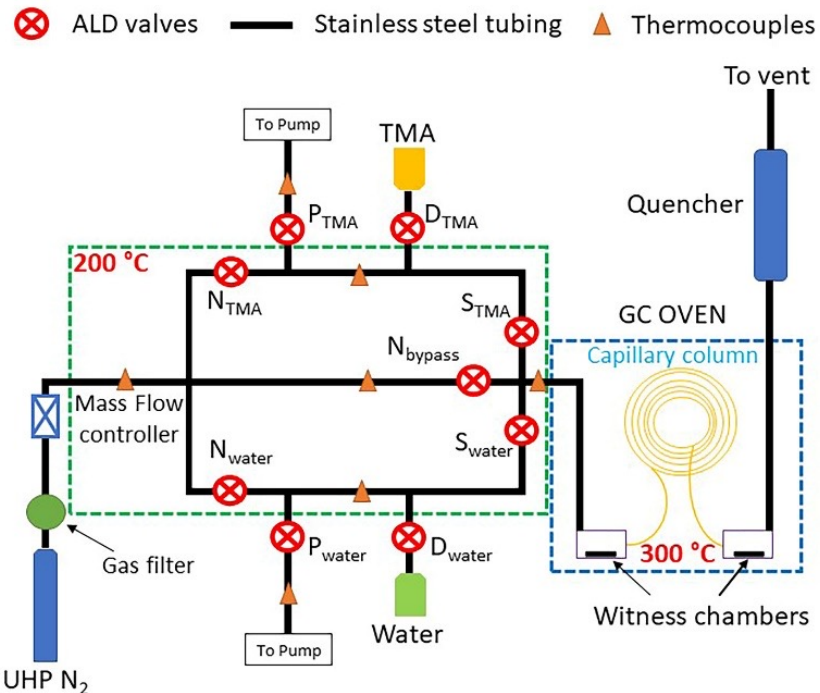
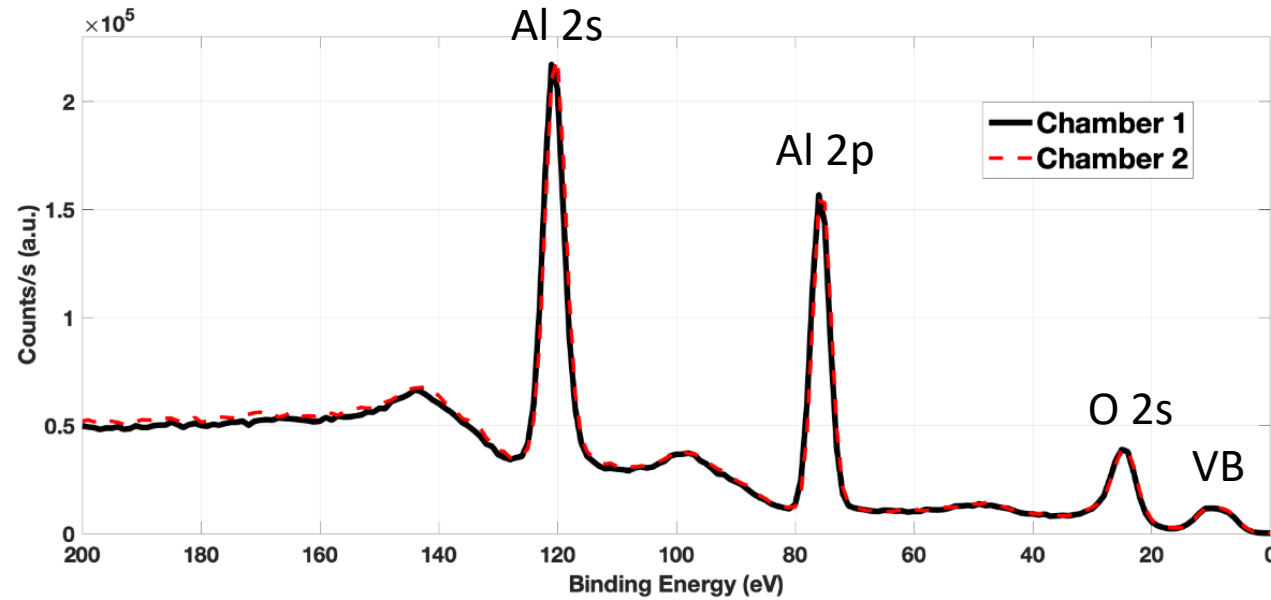
ALD of Alumina Monitored by SE and TEM



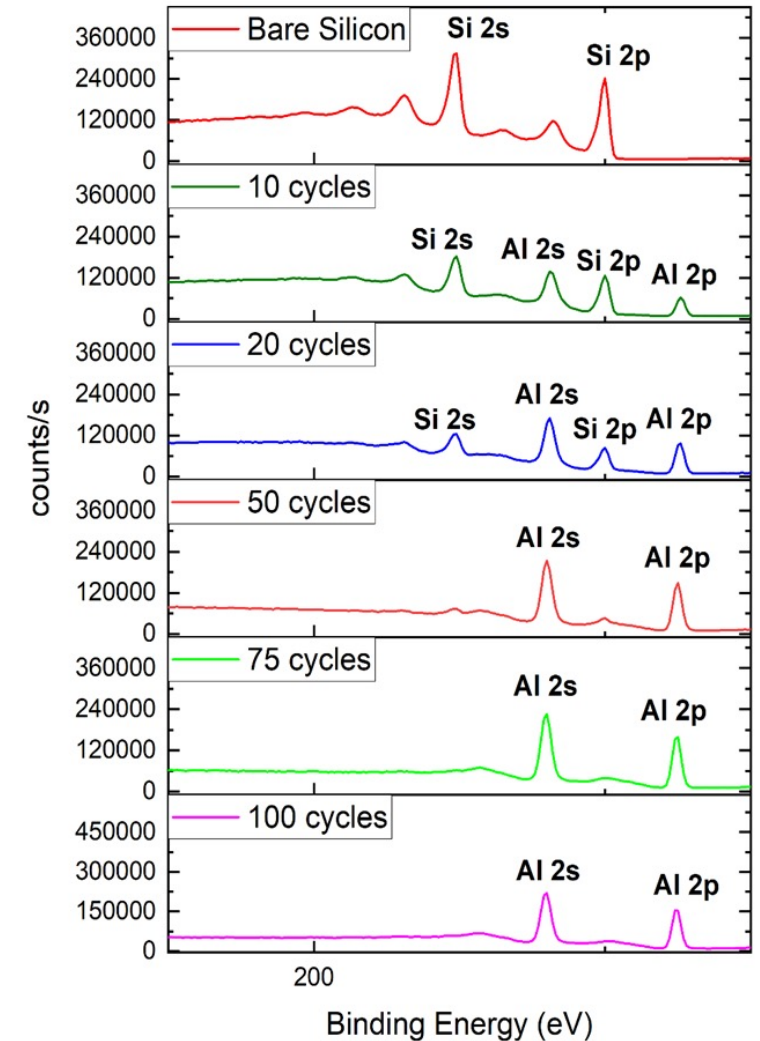
Conclusion: Basically the same thickness in the capillary and on the witness shards

A 5 m capillary was between the chambers in these SE and XPS experiments.

Characterization by XPS



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Glass Surface Chemistry: The Silanol

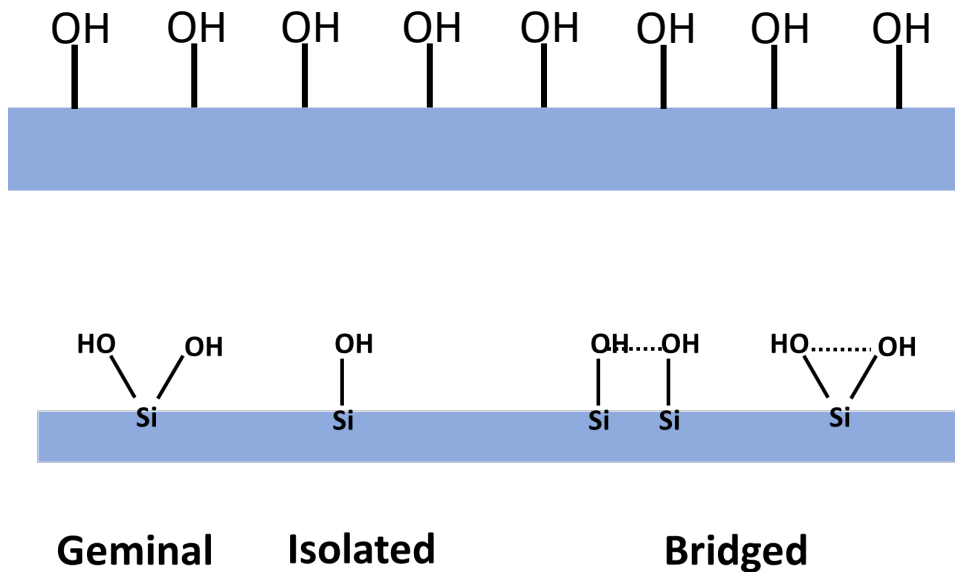


Figure 1. Schematic showing difference types Si-OH at the surface.

- The most important functional group on the glass surface is the silanol (Si-OH)
- Different types of surface silanols
- The surface density of silanols affects:
 - Particulate adhesion
 - Surface contamination rate
 - Charge buildup and discharge
 - Surface conductivity
 - Surface reactivity
 - Chemical durability
 - Water adsorption
 - Thin film adhesion

Glass Surface Chemistry: The Silanol

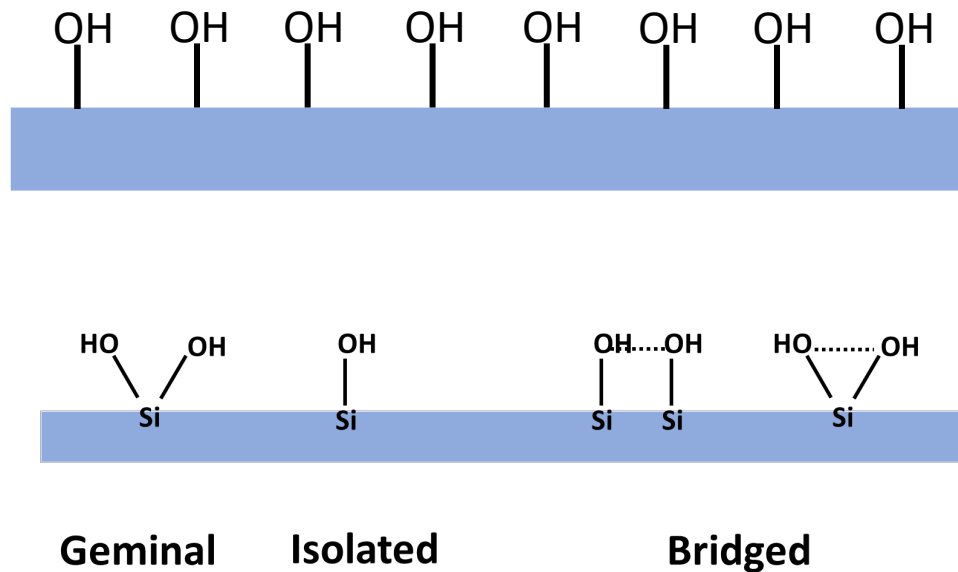


Figure 1. Schematic showing difference types Si-OH at the surface.

- A fully hydroxylated amorphous fused silica (SiO_2) surface has a silanol density of 4.6 OH/nm^2 (least squares average)
- Results from a combination of techniques (FTIR, XPS, ToF-SIMS, TGA, TPD-MS, BET) in different publications led to this number

Glass Surface Chemistry: The Silanol

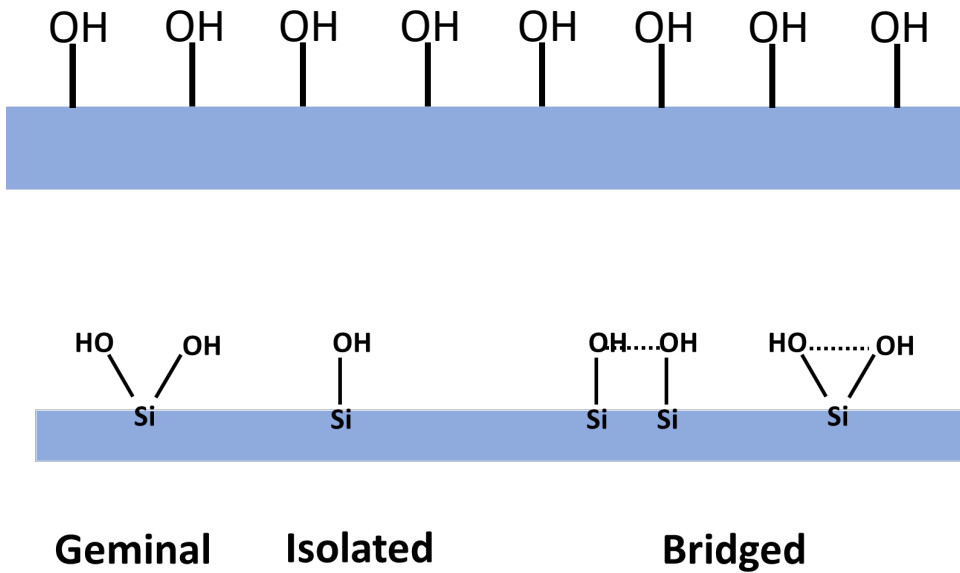
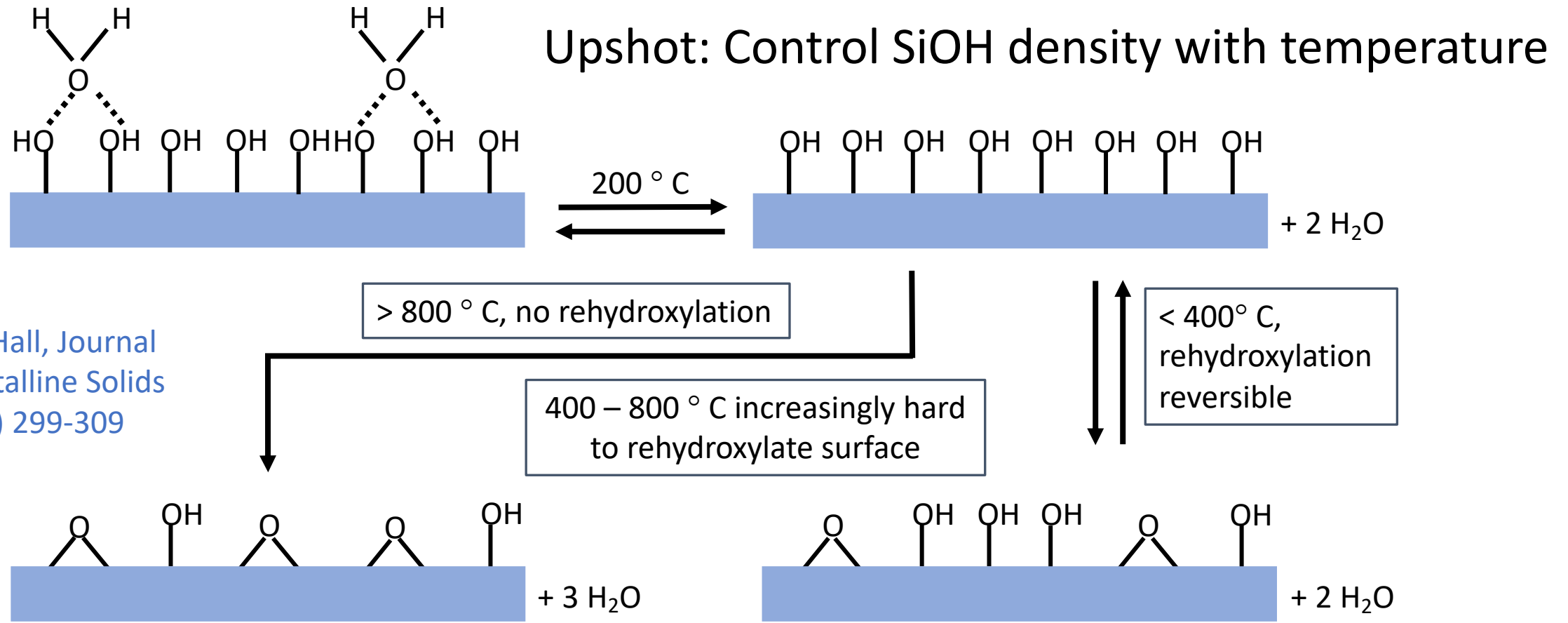


Figure 1. Schematic showing difference types Si-OH at the surface.

- People have quantified surface silanols by
 - Single-bounce FTIR
 - XPS
 - ToF- Secondary Ion Mass Spectrometry (ToF-SIMS)
- Problems:
 - Not completely sensitive to the outermost atomic layer of a material
 - Challenging to quantify results
- Possible Solution:
 - Tag SiOH groups to increase our sensitivity to them
 - How about with ALD?

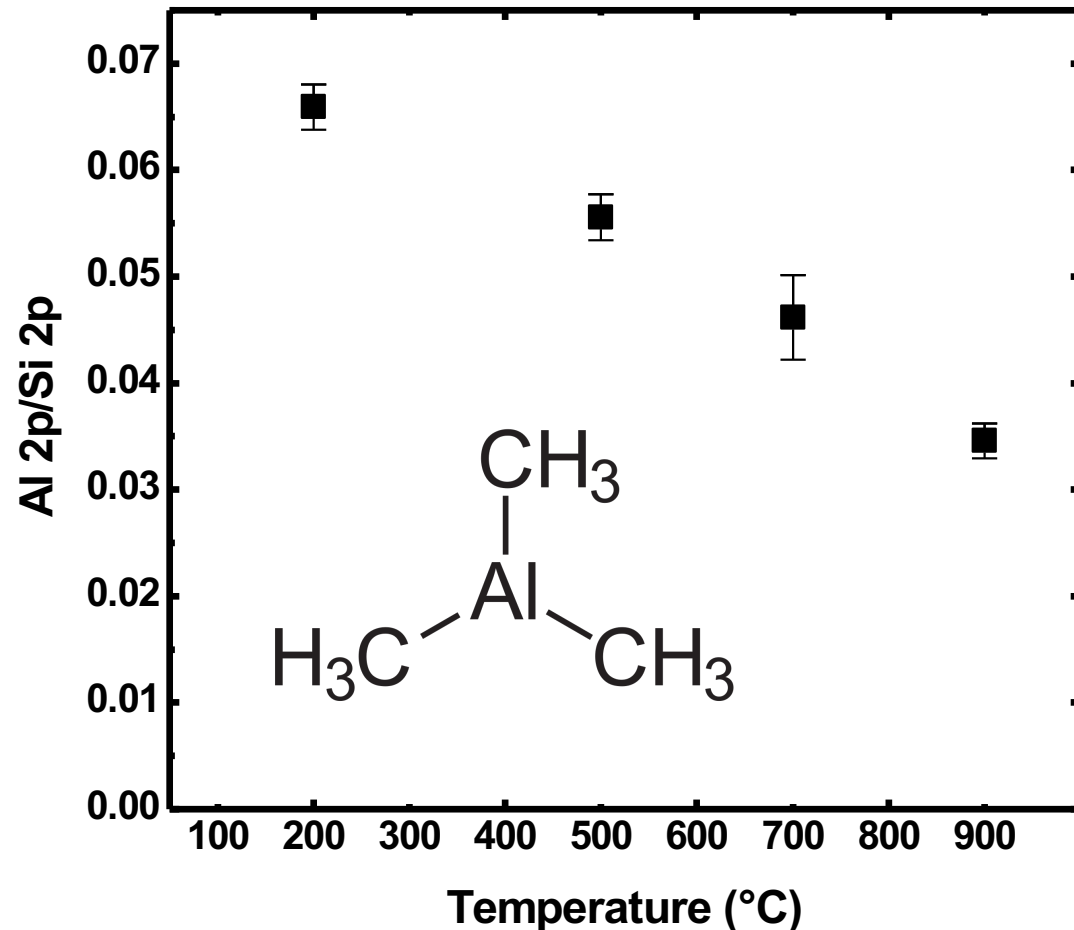
Dehydration and Dehydroxylation on SiO₂



Michael L. Hall, *Journal of Non-Crystalline Solids*
19 (1975) 299-309

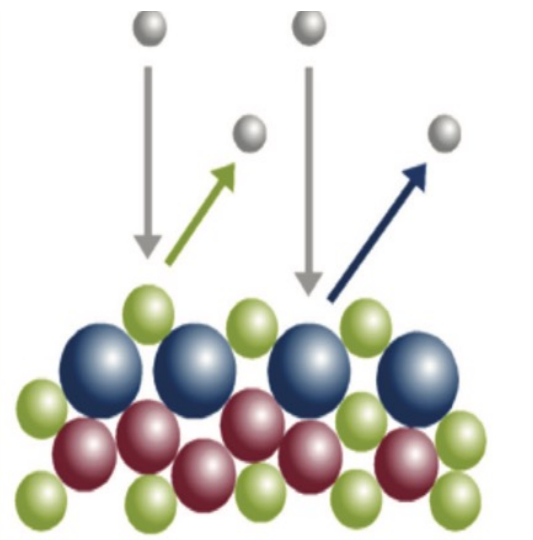
Figure 2. Schematic showing difference between Si-OH surface fully hydroxylated and after heat treatment

First Attempt: Tagging SiOH Groups with TMA – Single Layer ALD of Al



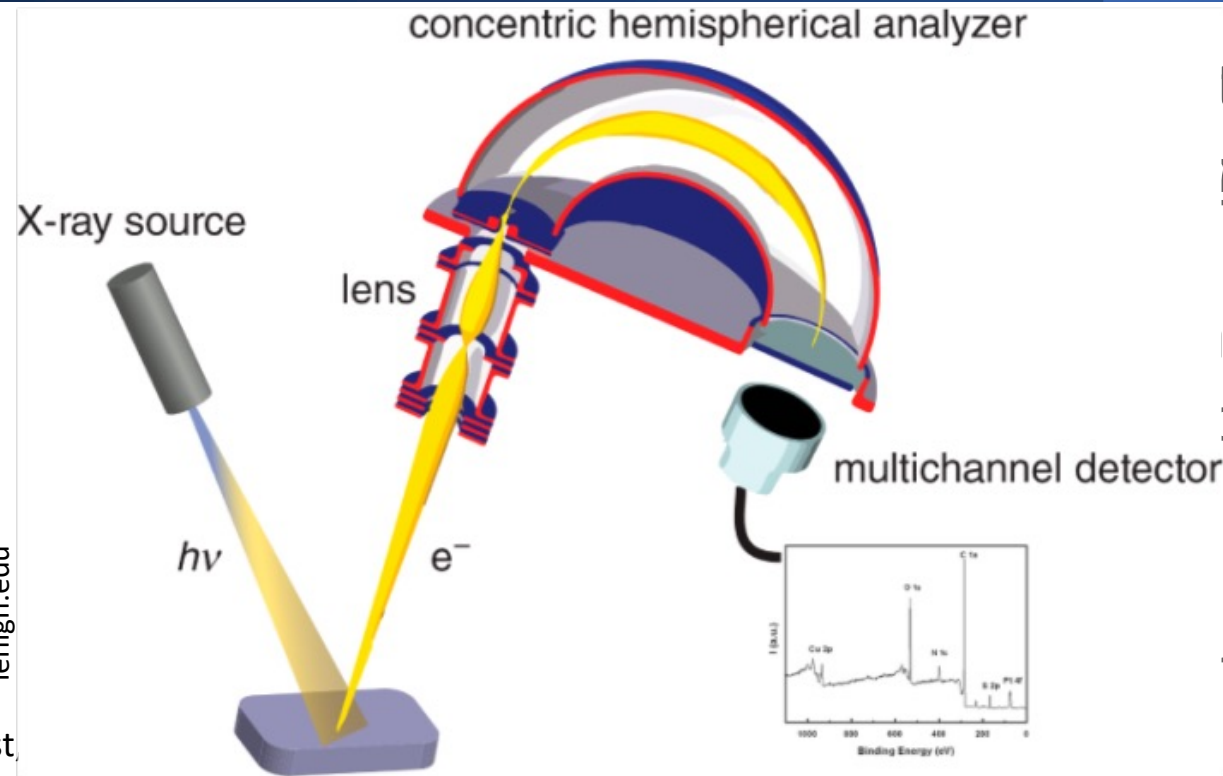
- XPS results challenging to quantify
- What about LEIS?
 - See next slide.

Low Energy Ion Scattering (LEIS) vs. ISS



Scattering of noble gas ions by surface atoms

lehigh.edu



to, and gives signal
most atomic layer of

ce with noble gas ions
noble gas ions is
keV

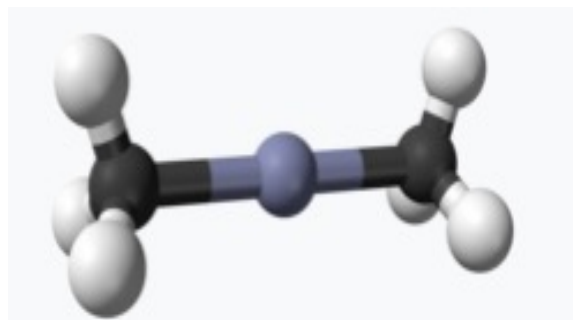
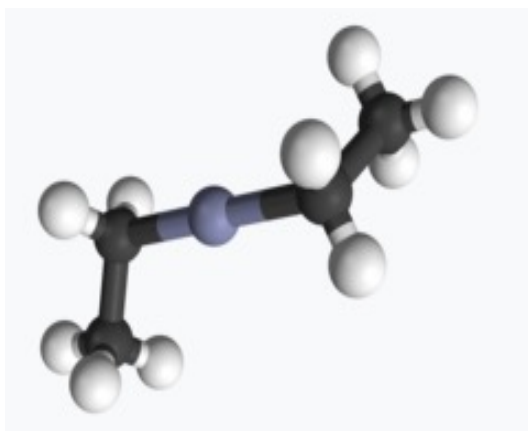
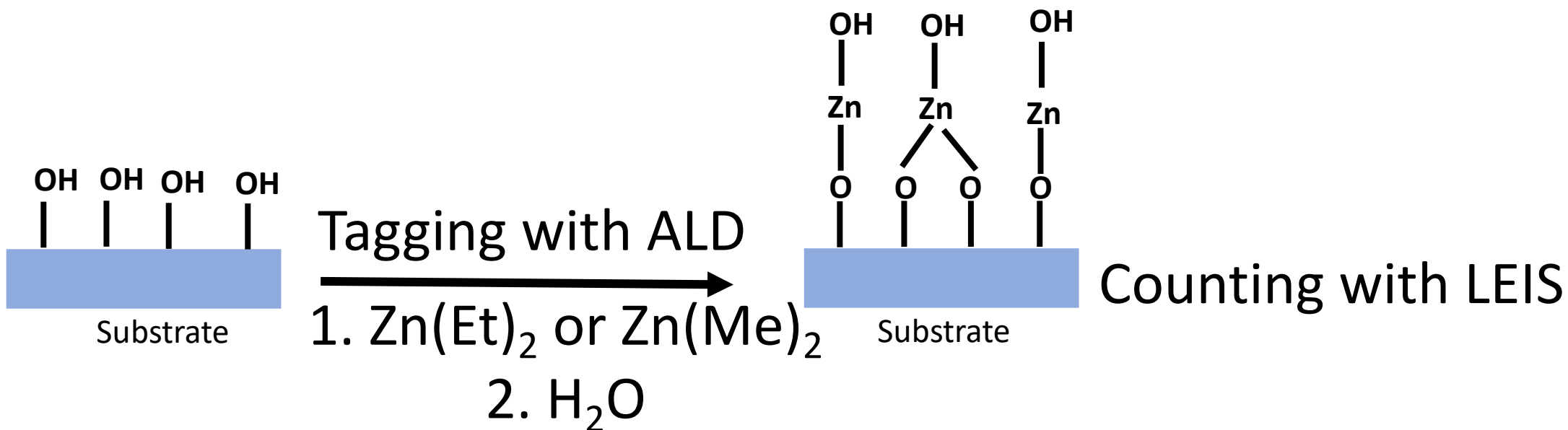
e backscattered ions
range for surface

- Cushman, Brüner, Zakel, Dahlquist, Linford "Low Energy Ion Scattering (LEIS) of As-Formed and Chemically Modified Display Glass: Significant Differences between Surface and Bulk Compositions as Revealed by LEIS Peak Fitting of the Al/Si Envelope" Applied Surface Science 2018, 455, 18 - 31.
- Cushman, Brüner, Zakel, Major, Lunt, Grehl, Smith, Linford "Low Energy Ion Scattering (LEIS). A Practical Introduction to its Theory, Instrumentation, and Applications." Analytical Methods, 2016, 8, 3419 – 3439.

- Advantages of modern LEIS vs. ISS
- But hard to differentiate between Si and Al in LEIS. Another tag?

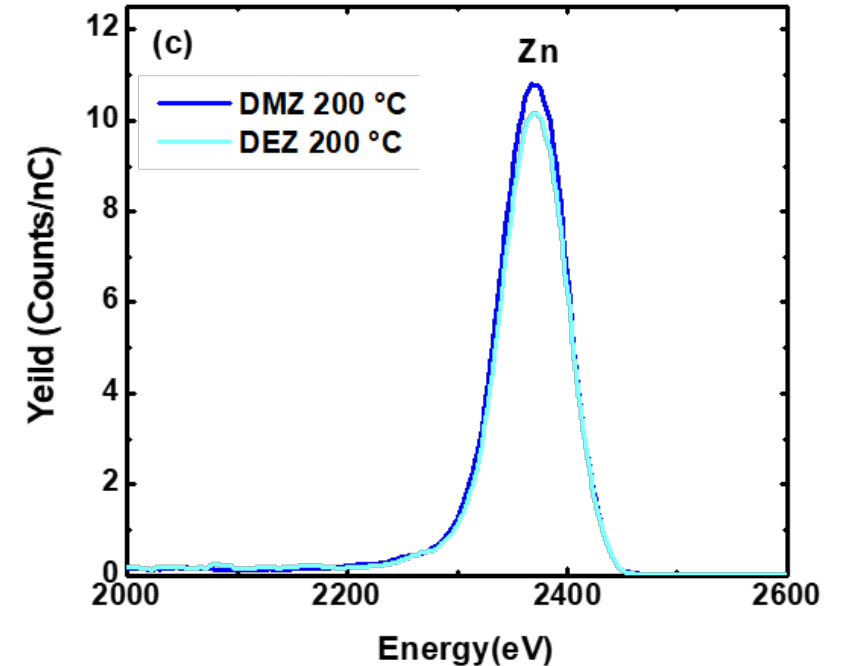
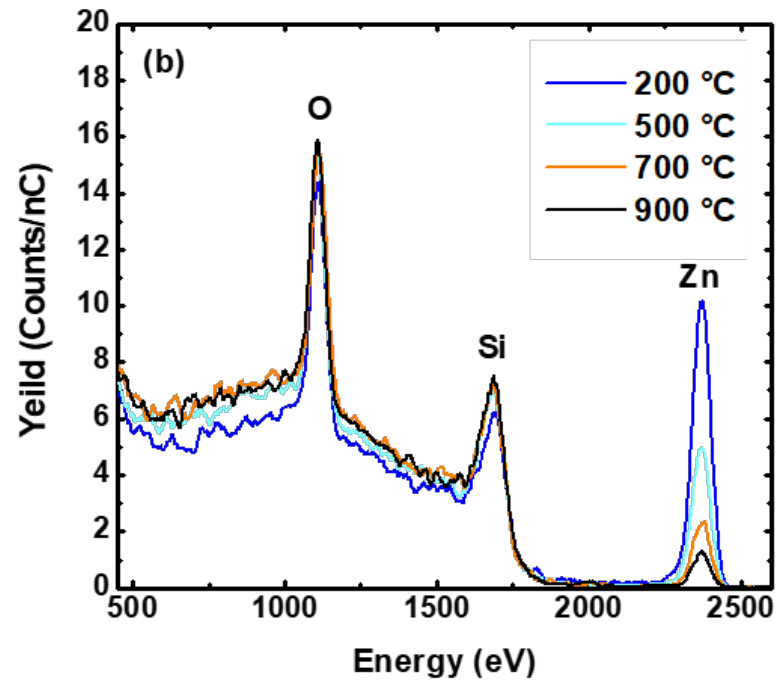
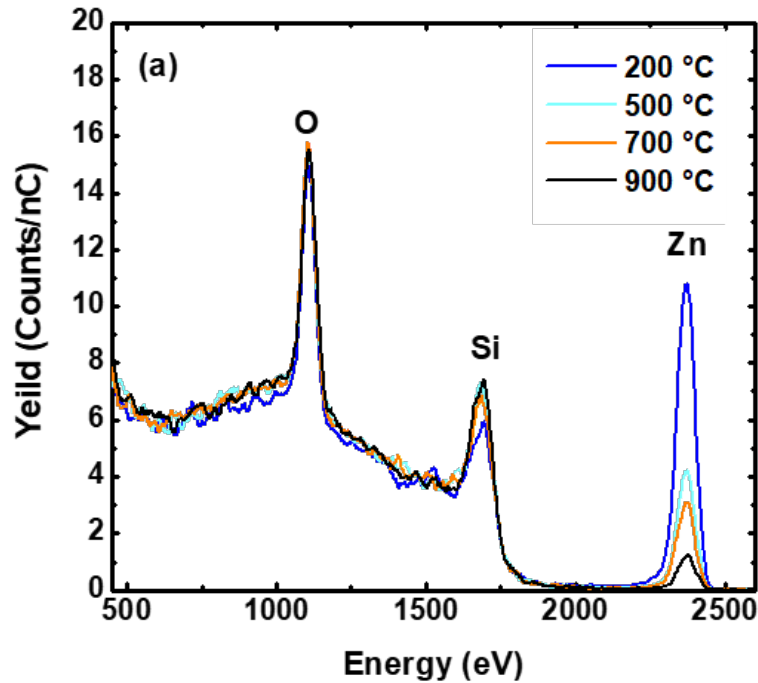
Tag and Count Approach

Gives a lower limit
to the number of
surface silanols



Avval, Průša, Cushman, Hodges, Fearn, Kim, Čechal, Vaníčková, Bábík, Šikola, Brongersma, Linford. "A Tag-and-Count Approach for Quantifying Surface Silanol Densities on Fused Silica Based on Atomic Layer Deposition and High-Sensitivity Low-Energy Ion Scattering." *Applied Surface Science* **2023**, 607, 154551.

LEIS of Zn-Tagged Silica Surfaces



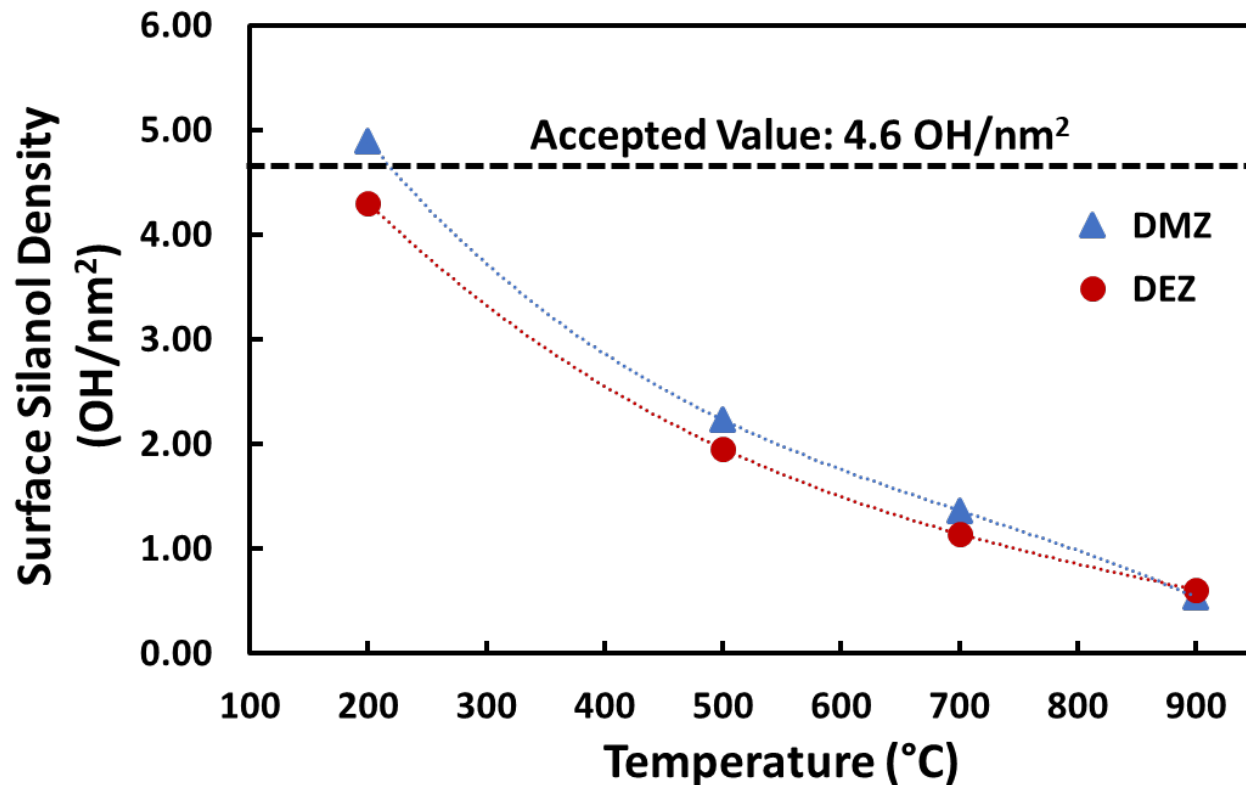
LEIS spectra of fused silica surfaces treated with single doses of (a) DMZ and water and (b) DEZ and water. (c) Comparison of the zinc signals for the fused silica samples treated at 200 °C in (a) and (b) and tagged with DMZ or DEZ. All the samples were cleaned with AO, and then heated to 270 °C prior to measurement with 3 keV He.

Quantifying the Number of Surface Silanols

$$\text{SiOH/nm}^2 = \frac{Zn_i}{Zn_{ref}} * \sigma_{ZnO} \left(\frac{\text{atom}}{\text{nm}^2} \right)$$

- $\left(\frac{Zn_i}{Zn_{ref}} \right)$ the surface coverage
- σ_{ZnO} : 10.9 ZnO units/nm²
 - Based on literature value for the density of ZnO: 5.61 g/cm³

Quantifying the Number of Surface Silanols



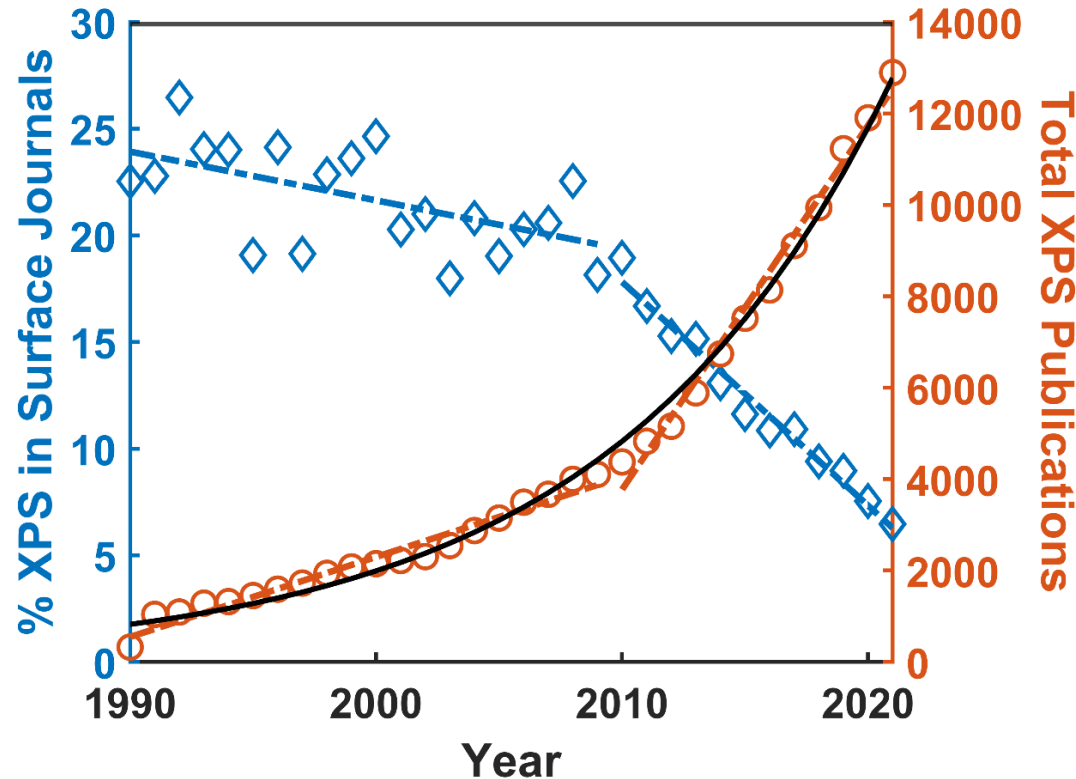
- Actual quantitation!
- Results from both precursors within band in Zhuravlev review!
- Significant contrast for samples treated at different temperatures
- Consistent small differences between the two precursors
- Isn't based on knowing the right answer to start with

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XPS is Growing, but Being Used Less by Experts

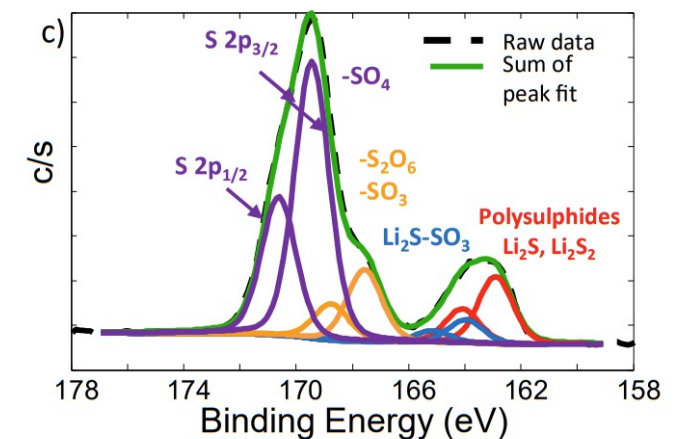
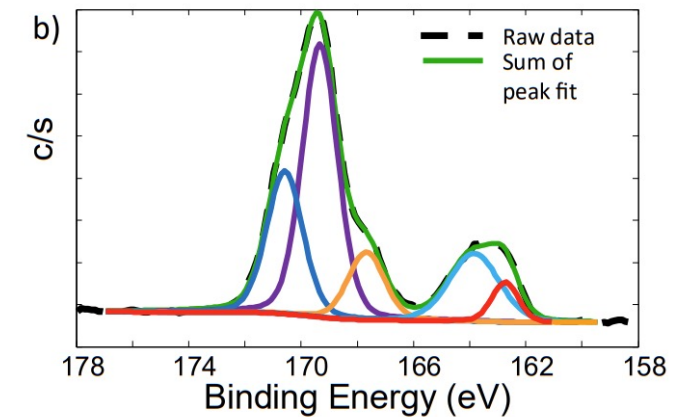
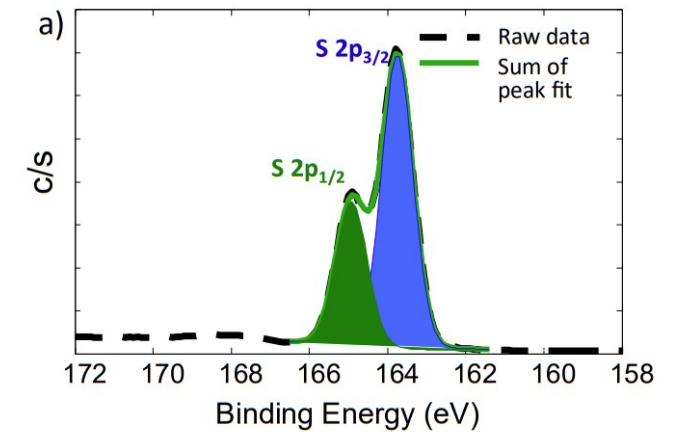


- The literature is increasingly filled with bad XPS peak fitting
 - Let's look at some examples

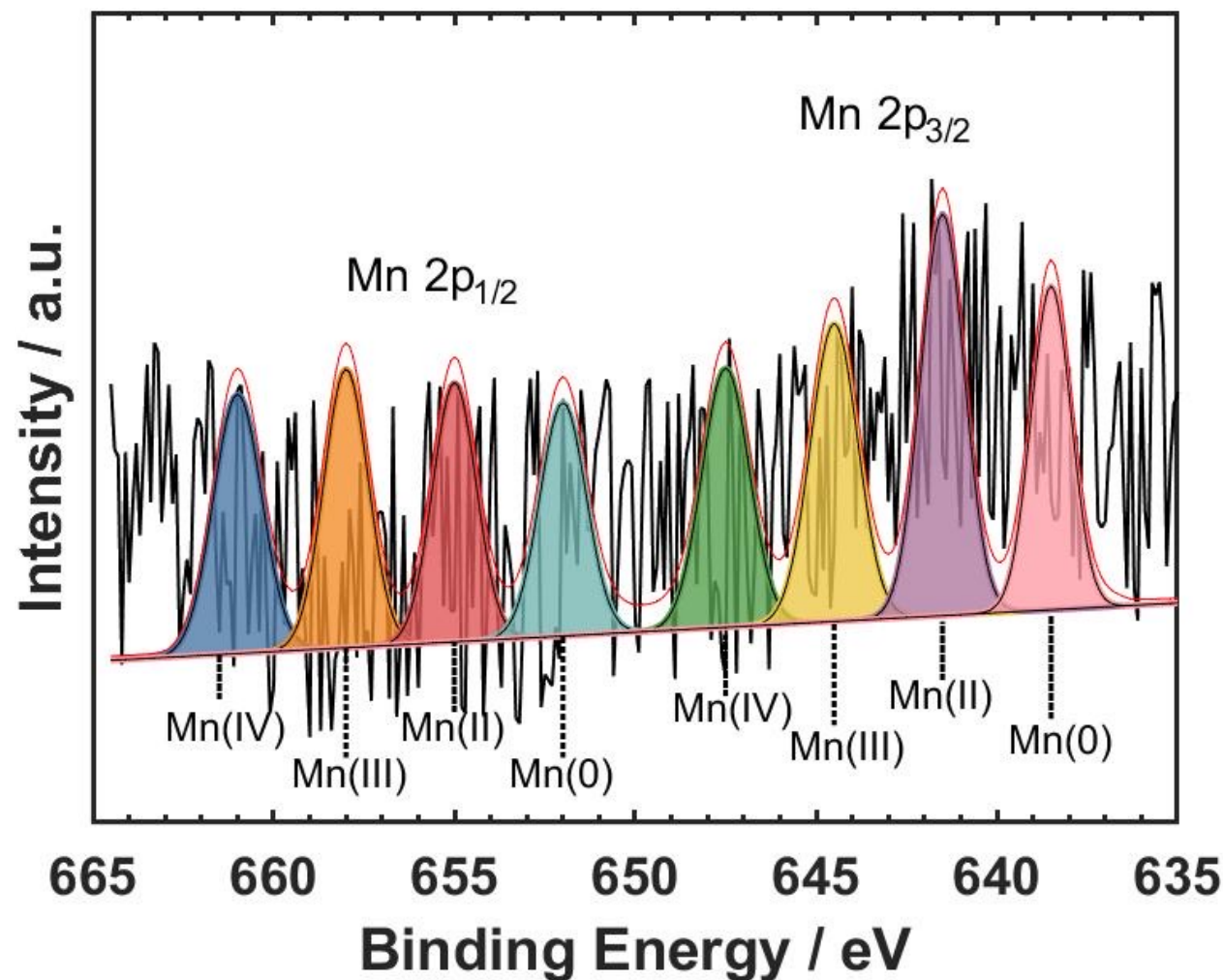
- Web of Science search on titles, abstracts, and keywords for 'XPS' by year.
- 13 established surface journals
 - Journal of Vacuum Science and Technology A
 - Journal of Vacuum Science and Technology B
 - Surface Science Spectra
 - Langmuir
 - Surface and Interface Analysis
 - Journal of Physical Chemistry C
 - Surface Science
 - Applied Surface Science
 - Surface and Coatings Technology
 - Thin Solid Films
 - Colloids and Surfaces B
 - Surface Review and Letters
 - Progress in Surface Science

Examples of Bad XPS Peak Fitting in the Literature or in Presentations

- Concept of spin-orbit splitting
- *s* orbitals show one peak
- *p*, *d*, *f* orbitals show 2 peaks
- 2:1, 3:2, 4:3 area ratios

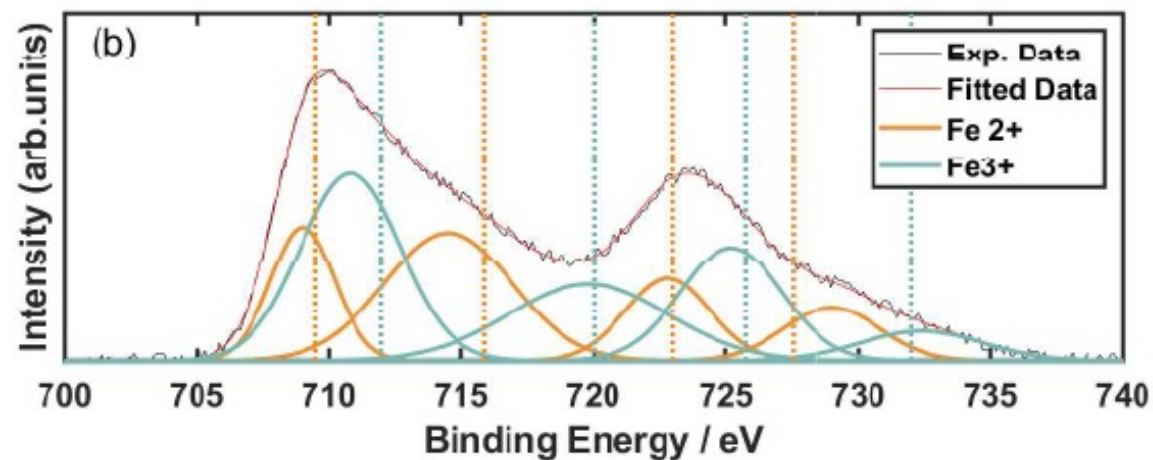
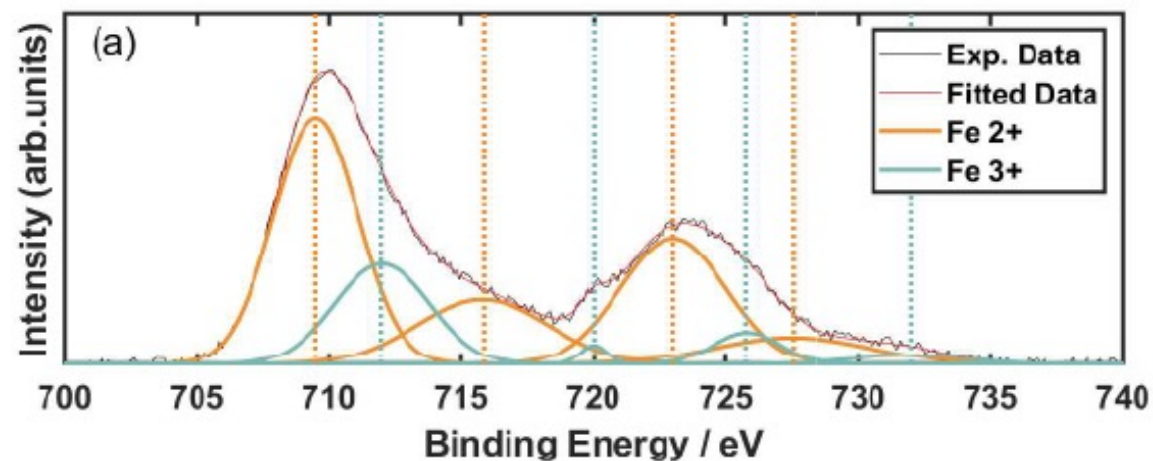


Examples of Bad XPS Peak Fitting in the Literature or in Presentations

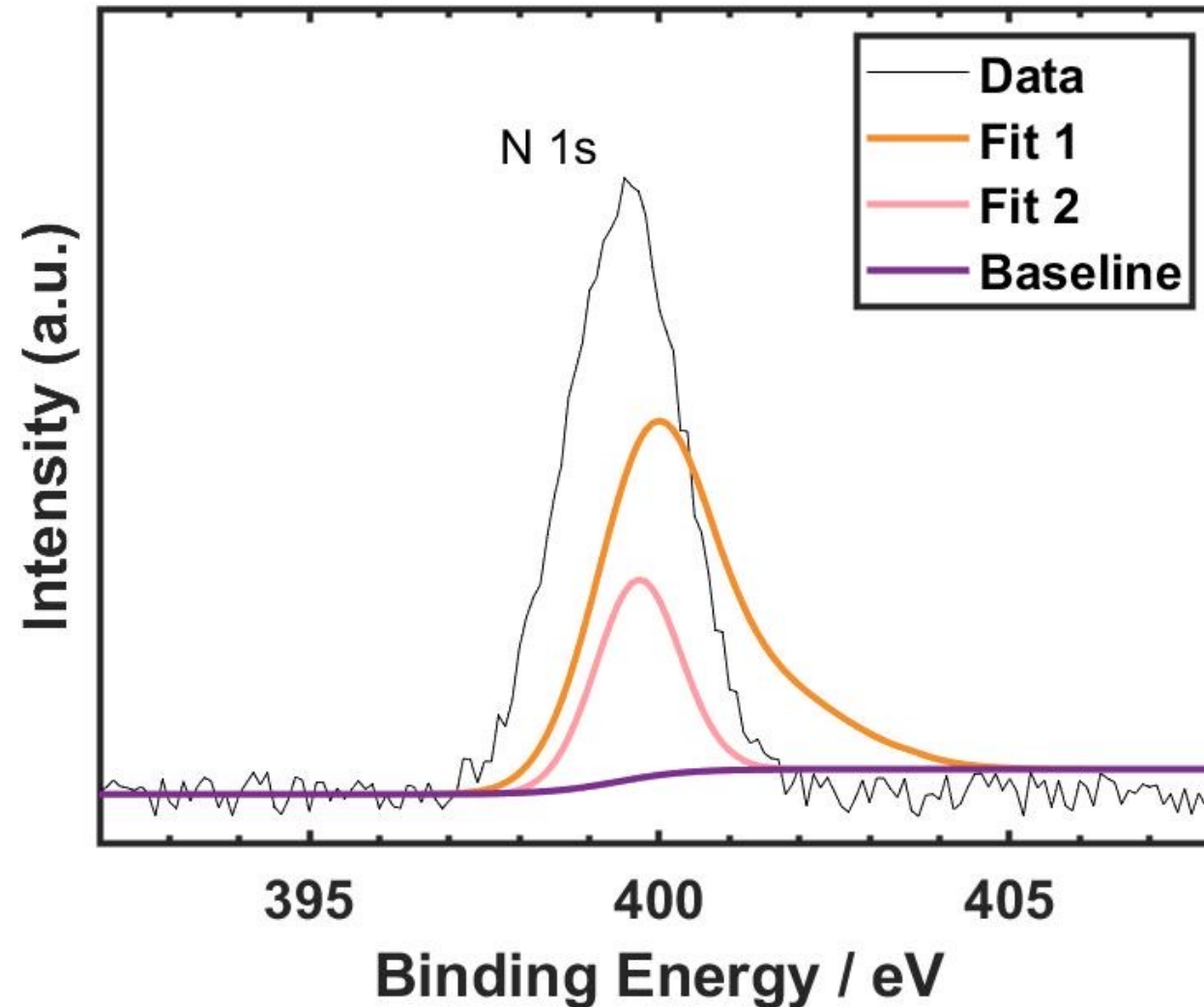


Examples of Bad XPS Peak Fitting in the Literature or in Presentations

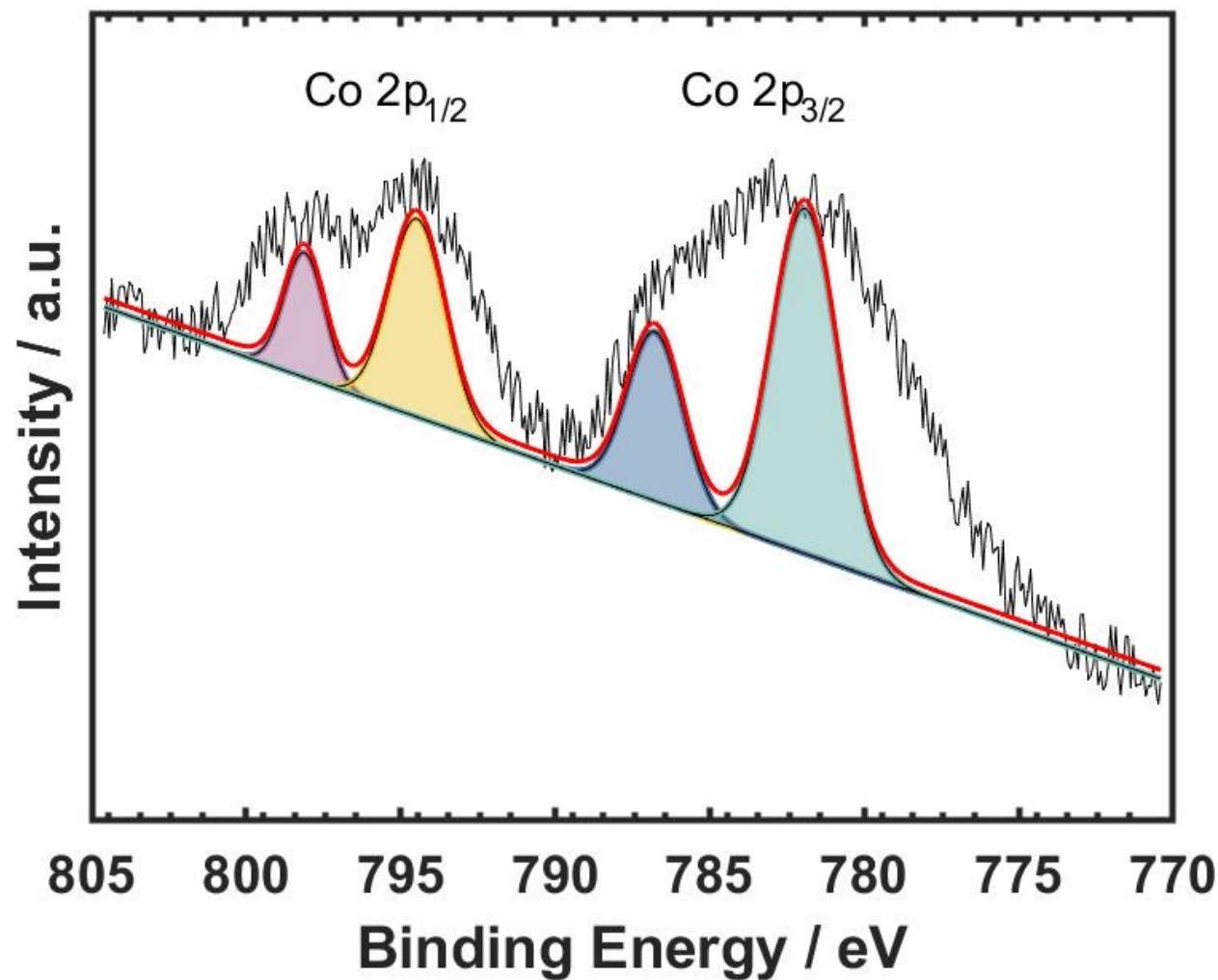
- Fe 2p region



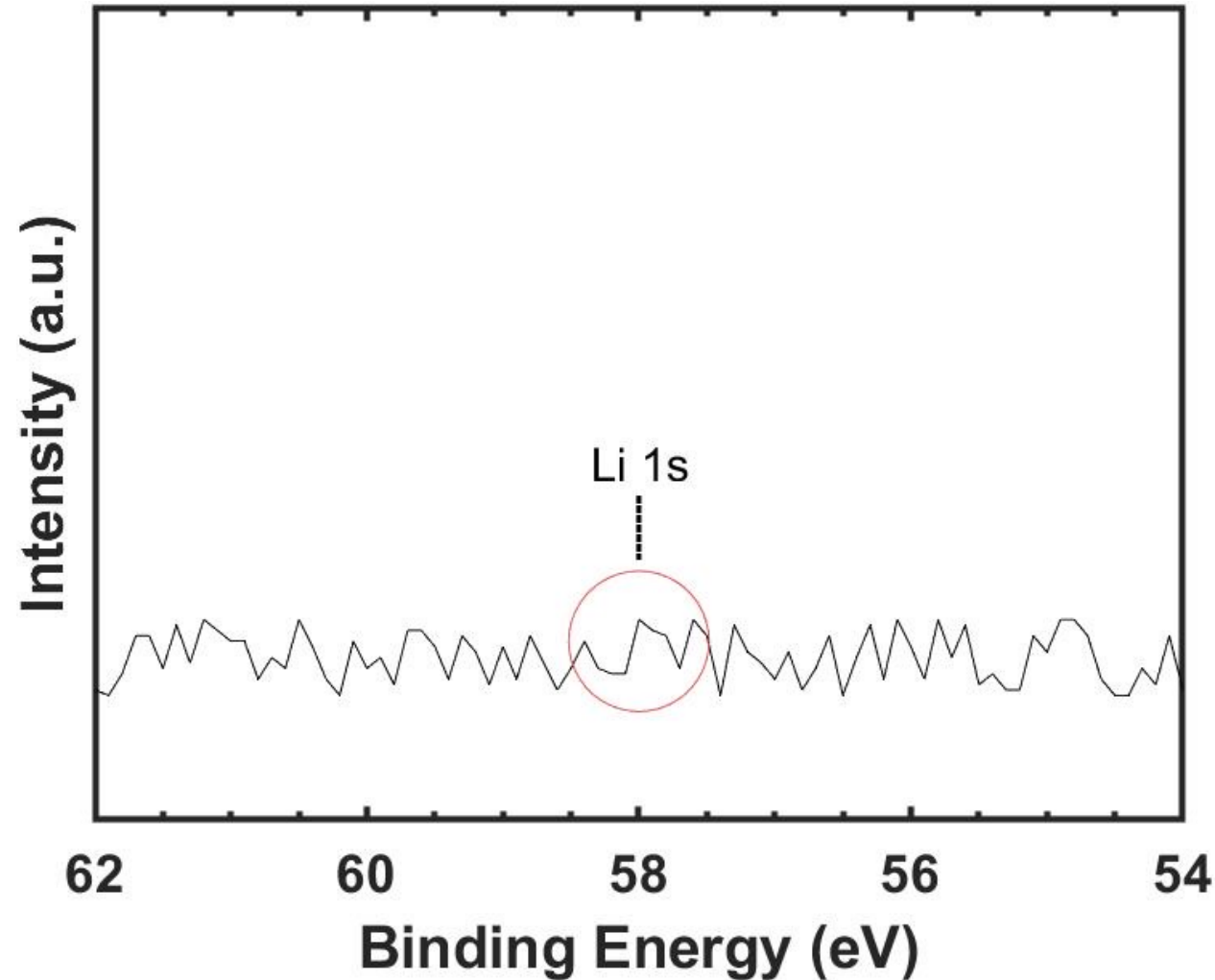
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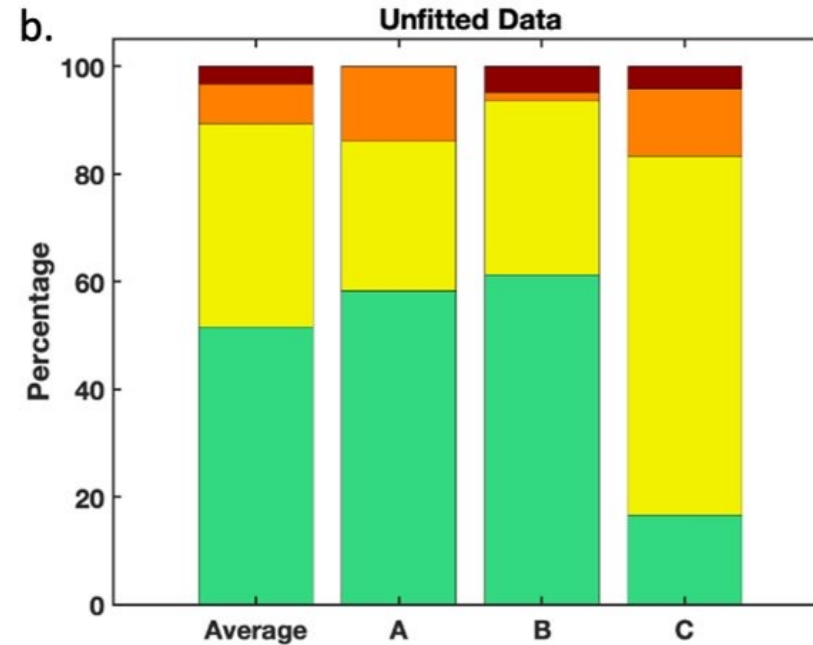


Examples of Bad XPS Peak Fitting in the Literature or in Presentations



Quantifying the problem

ca. 400 papers in 3 high-quality journals during 2019

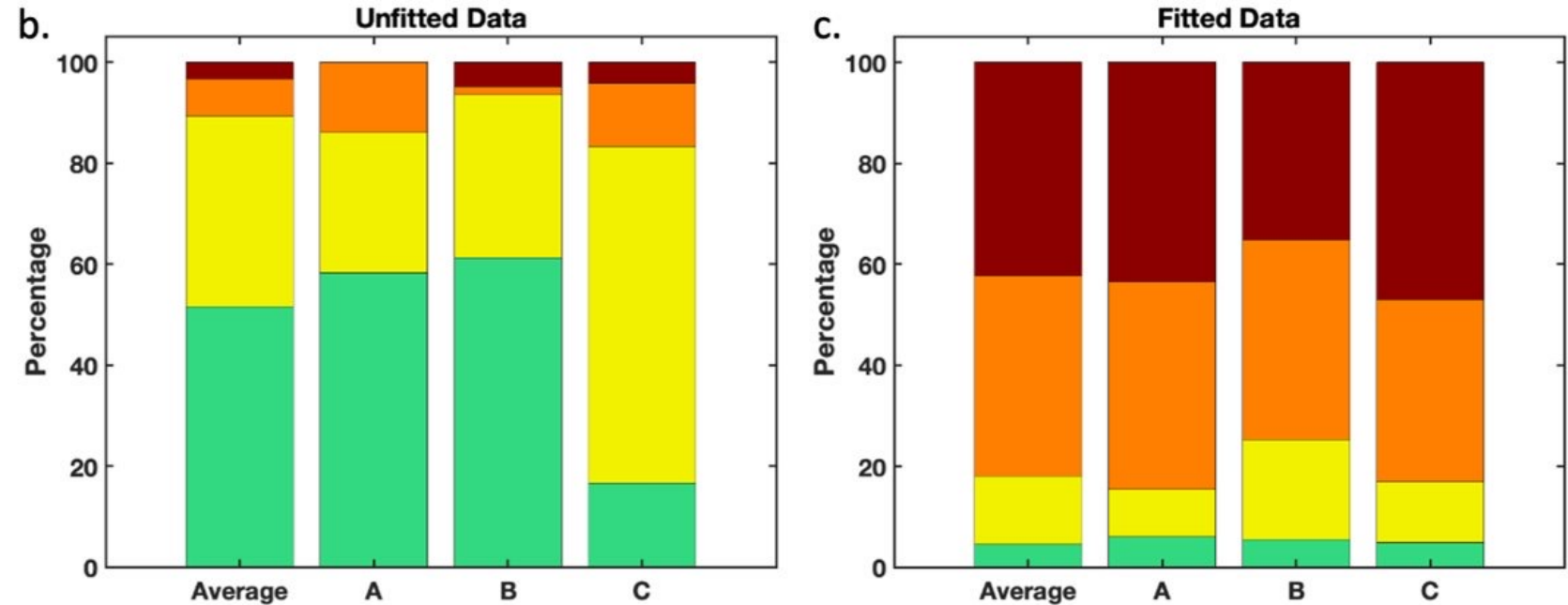


METHODOLOGY:

George H. Major, Tahereh G. Avval, Behnam Moeini, Gabriele Pinto, Dhruv Shah, Varun Jain, Victoria Carver, **William Skinner, Thomas R. Gengenbach, Christopher D. Easton, Alberto Herrera-Gomez**, Tim S. Nunney, **Donald R. Baer, Matthew R. Linford** 'An Assessment of the Frequency and Nature of Erroneous X-ray Photoelectron Spectroscopy (XPS) Analyses in the Scientific Literature'. *J. Vac. Sci. Technol. A* **2020**, 38, 061204. DOI: 10.1116/6.0000685.

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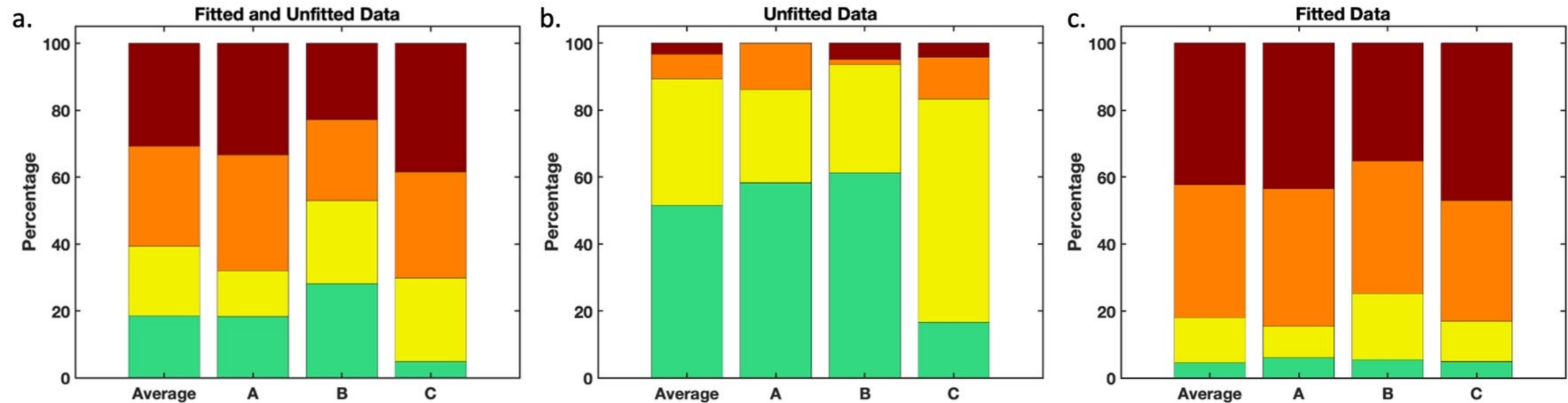


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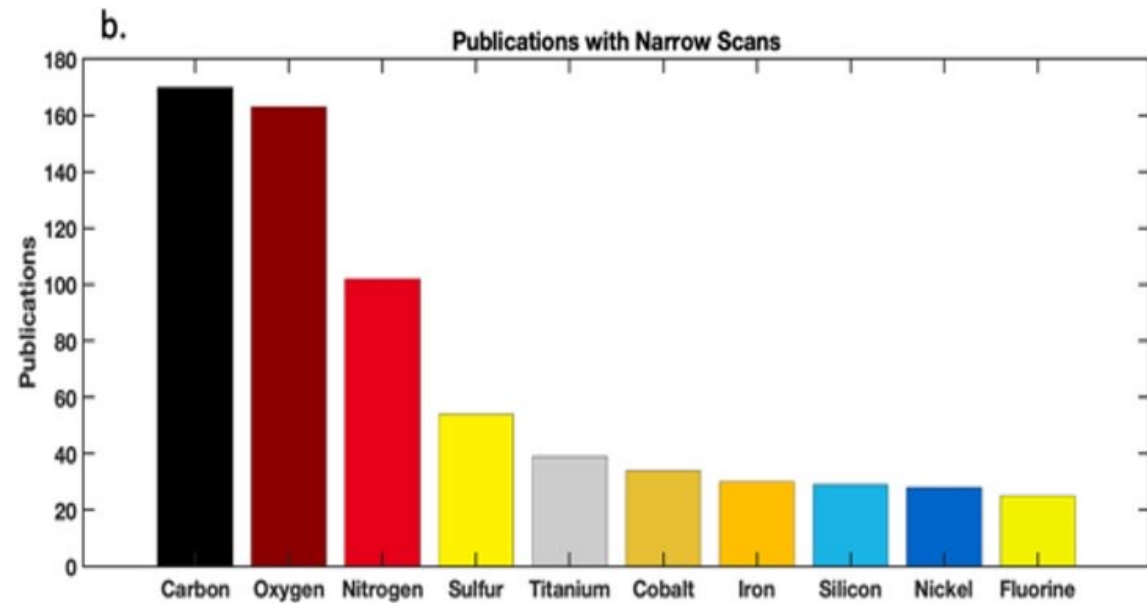
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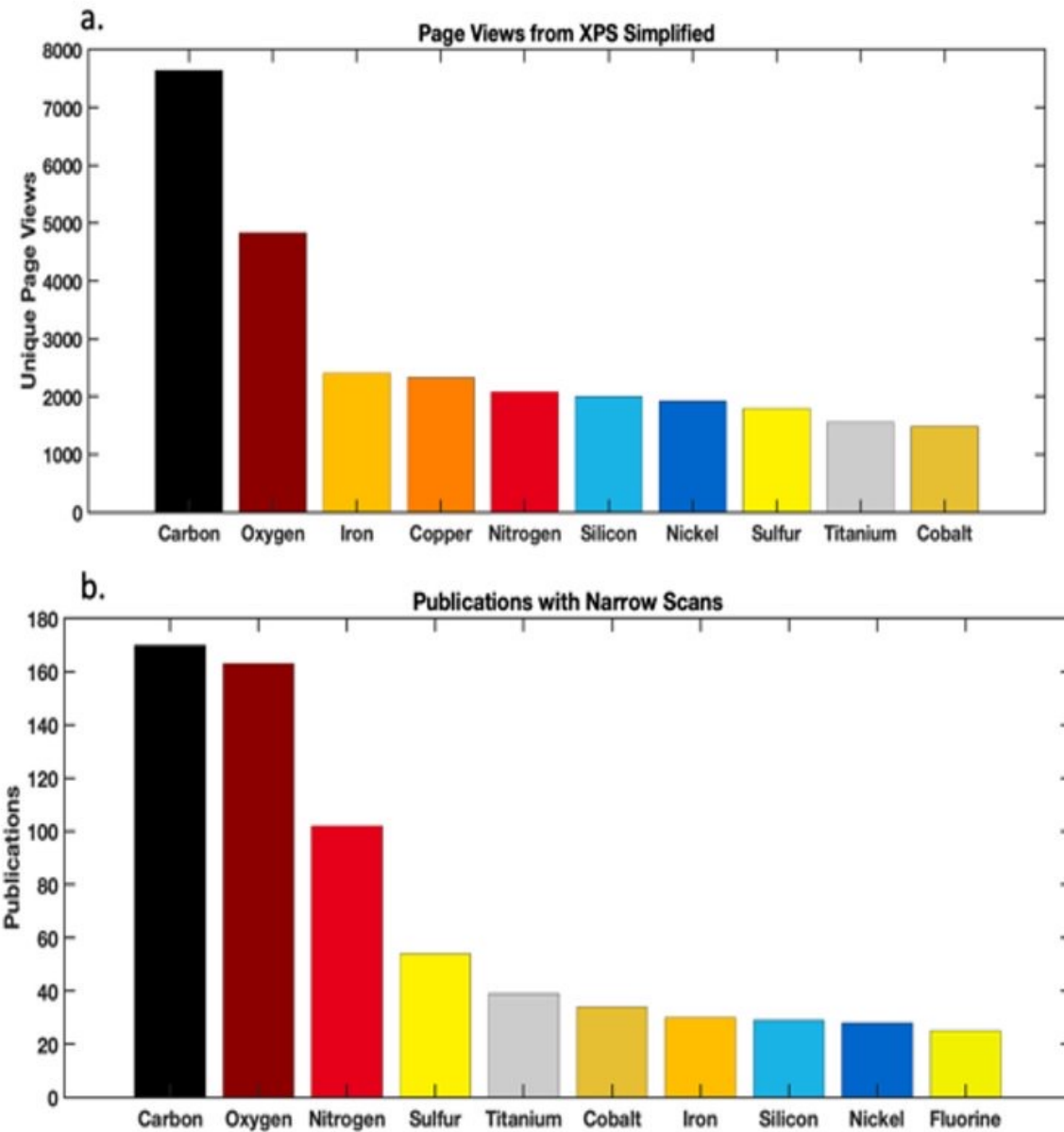
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Other results
from this study
– elements
from which
elements are
most often fit?



1 H	Click an element for XPS information																2 He				
3 Li	4 Be															5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg															13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr				
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe				
55 Cs	56 Ba	57 La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu					
87 Fr	88 Ra	89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr					

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Guide to Peak Fitting C 1s Spectra

Google Scholar:
138 Citations

Practical guides for x-ray photoelectron spectroscopy (XPS): Interpreting the carbon 1s spectrum

Cite as: J. Vac. Sci. Technol. A 39, 013204 (2021); doi: 10.1116/6.0000682

Submitted: 30 September 2020 · Accepted: 7 December 2020 ·

Published Online: 6 January 2021



Thomas R. Gengenbach,^{1,a)} George H. Major,² Matthew R. Linford,^{2,a)} and Christopher D. Easton^{1,a)}

AFFILIATIONS

¹Commonwealth Scientific and Industrial Research Organisation (CSIRO) Manufacturing, Clayton, Victoria 3168, Australia

²Department of Chemistry and Biochemistry, Brigham Young University, Provo, Utah 84602

Note: This paper is part of the Special Topic Collection on Reproducibility Challenges and Solutions.

Electronic addresses: thomas.gengenbach@csiro.au, chris.easton@csiro.au, mrlinford@chem.byu.edu

ABSTRACT

The carbon 1s photoelectron spectrum is the most widely fit and analyzed narrow scan in the x-ray photoelectron spectroscopy (XPS) literature. It is, therefore, critically important to adopt well-established protocols based on best practices for its analysis, since results of these efforts affect research outcomes in a wide range of different application areas across materials science. Unfortunately, much XPS peak fitting in the scientific literature is inaccurate. In this guide, we describe and explain the most common problems associated with C 1s narrow scan analysis in the XPS literature. We then provide an overview of rules, principles, and considerations that, taken together, should guide the approach to the analysis of C 1s spectra. We propose that following this approach should result in (1) the avoidance of common problems and (2) the extraction of reliable, reproducible, and meaningful information from experimental data.

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I. INTRODUCTION

Carbon is not particularly abundant in the Earth's crust. With an estimated concentration of several hundred to thousand parts per million (ppm) by weight, it does not even rank among the top ten most abundant elements. However, it is ubiquitous at the Earth's surface, i.e., the human environment, in solid, liquid, or gaseous form. In spite of carbon's relatively low overall abundance, its importance cannot be overstated. Unlike most elements, carbon readily bonds to itself to form stable single, double, and triple bonds, forming chains and rings, and combining with other elements in a way that produces an essentially infinite number of organic compounds with molecular weights that range from little more than a dozen to millions of atomic mass units (amu or g/mol). Accordingly, it is rare in the day-to-day work of an analytical laboratory to study samples that do not contain carbon in some form. This is particularly true for surface analytical laboratories that use highly surface-sensitive techniques such as x-ray photoelectron spectroscopy (XPS) to characterize materials. This carbon at sample surfaces (or in the bulk in an XPS depth profile) has many potential sources:

- The material itself may be carbonaceous. Such materials include graphitic carbon, e.g., graphene and carbon nanotubes; organic materials, such as many polymers and self-assembled monolayers; and diamond (and doped diamond).
- The material may be a hybrid of inorganic and organic constituents, either by nature or by design, e.g., metal organic frameworks and carbon steel.
- Organic impurities, either in the bulk or as surface contamination, the latter typically known as adventitious carbon (AdC).

The presence of carbon in XPS analyses is both directly and indirectly felt. Carbon may perturb (chemically shift) the peak positions of other elements to which it is bonded. The valence band signals from carbon-containing surfaces/materials, i.e., the signals at binding energies (BE) of ~0–30 eV, are a direct result of carbon in chemical bonds and often constitute a “fingerprint” for a material. Even if only to a small degree, AdC attenuates the XPS signals that originate below it. Carbon also produces a strong Auger signal, which is fairly broad but often ignored because of its complexity. Nevertheless, in spite of the usefulness, or potential

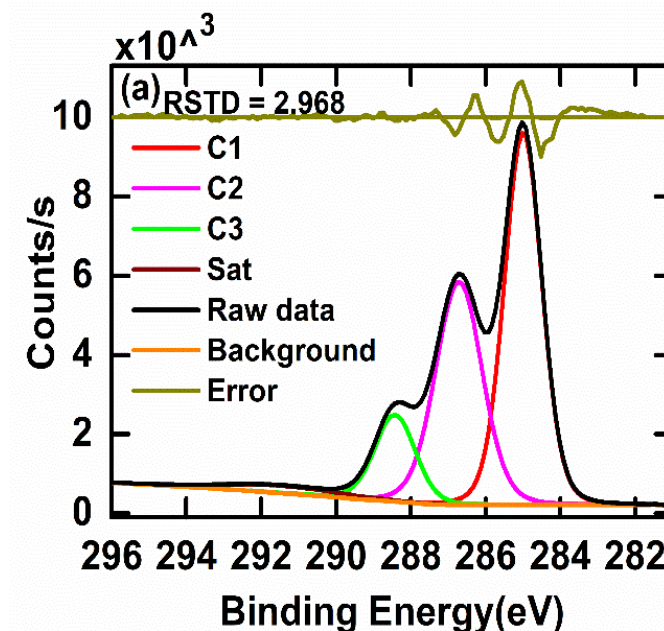
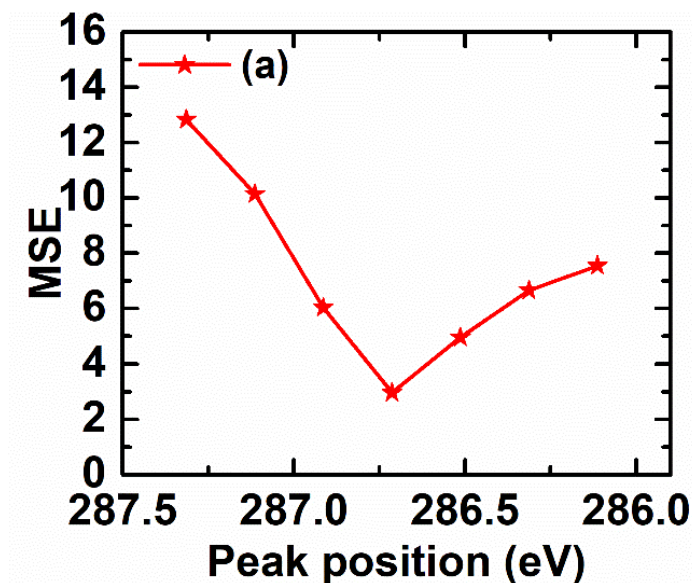
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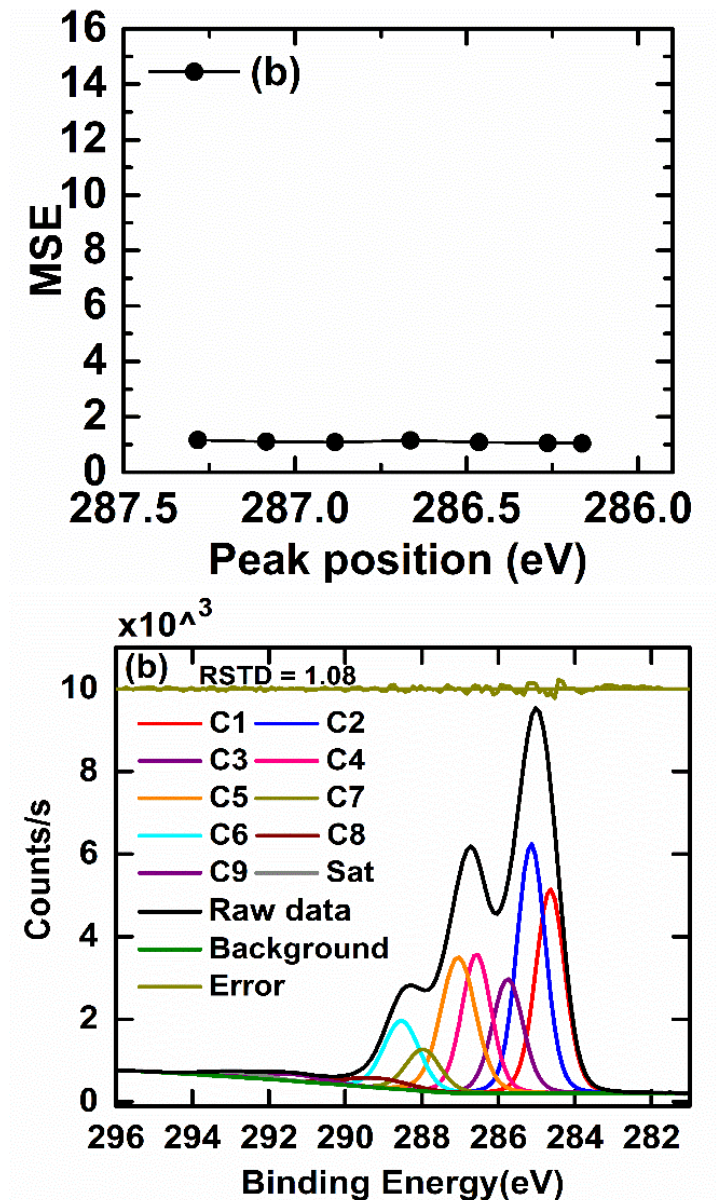
Uniqueness Plots

- Peak fitting is widely used in XPS data analysis
- Need methods for determining/estimating the statistical quality of the fits
- Uniqueness plots are widely used in spectroscopic ellipsometry
- Consistently change a part of the fit and see how the MSE changes
- Moved the 'C2' component here
- Does the fit have the ability to compensate for your changes?
- If it does, it isn't 'unique'. There is fit parameter correlation.
- Look for a 'U' or 'V' shape. An absence of fit parameter correlation.



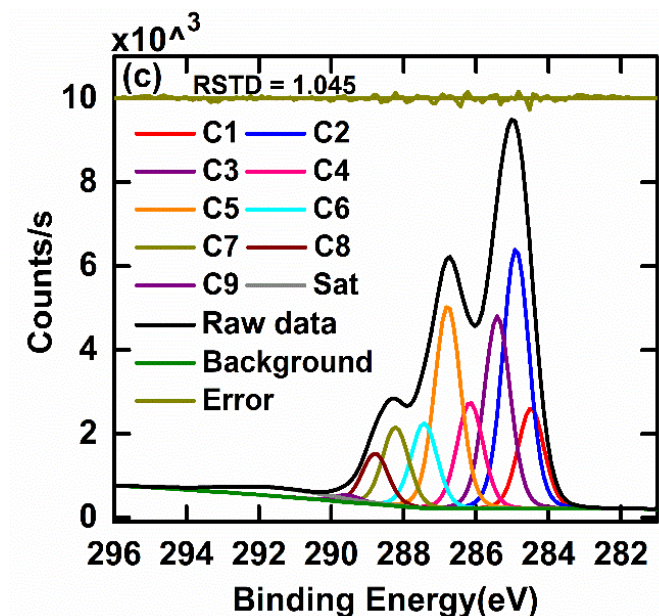
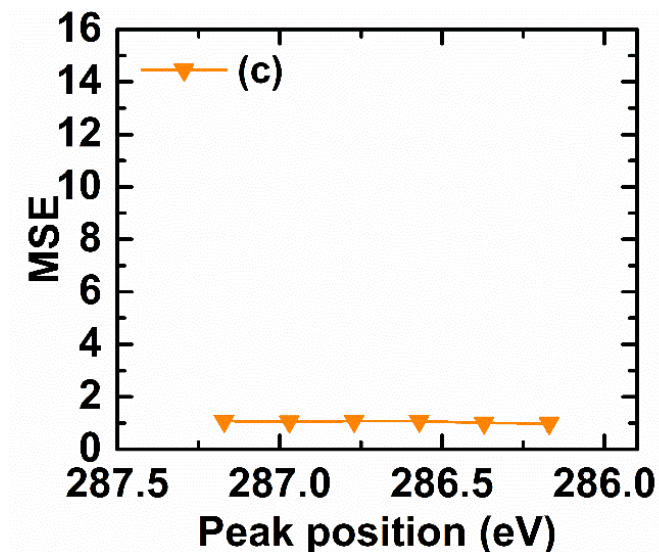
Uniqueness Plots

- Uniqueness plot of the 10-component fit with ***no constraints on the fitting parameters***
- Moved the 'C4' component here



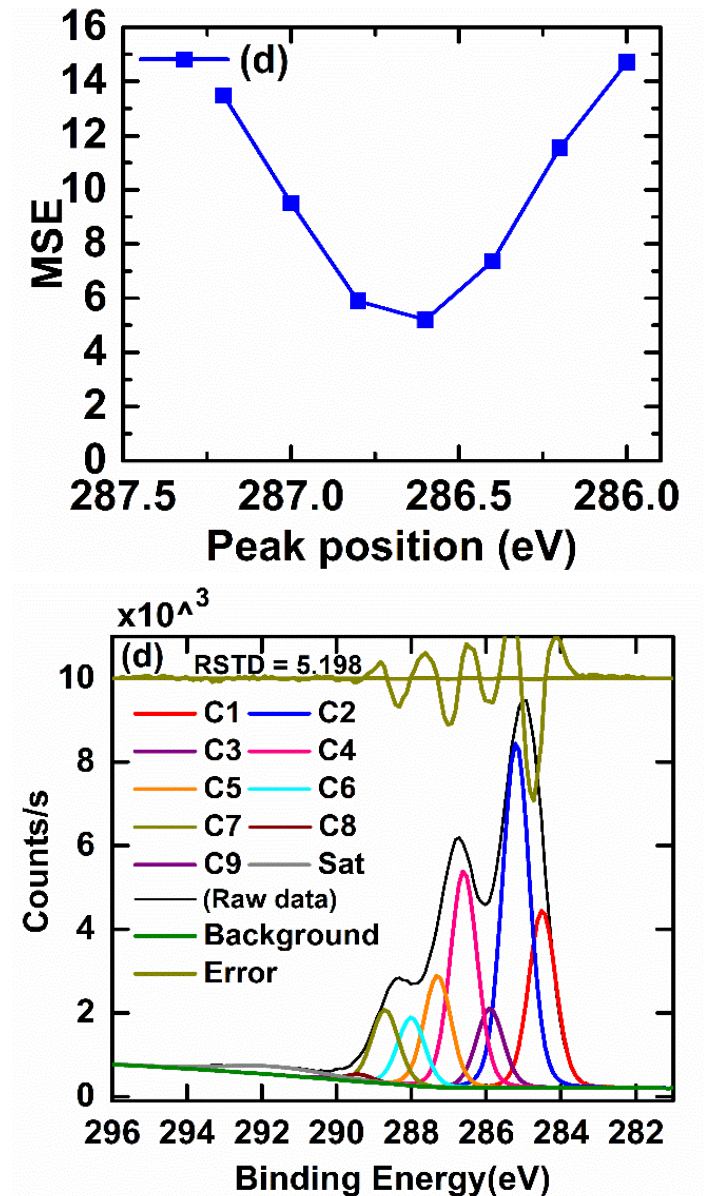
Uniqueness Plots

- Uniqueness plot of the 10-component fit
- ***The only constraint was that the nine peaks modeling the main peak envelope had the same widths***
- Moved the 'C4' component here



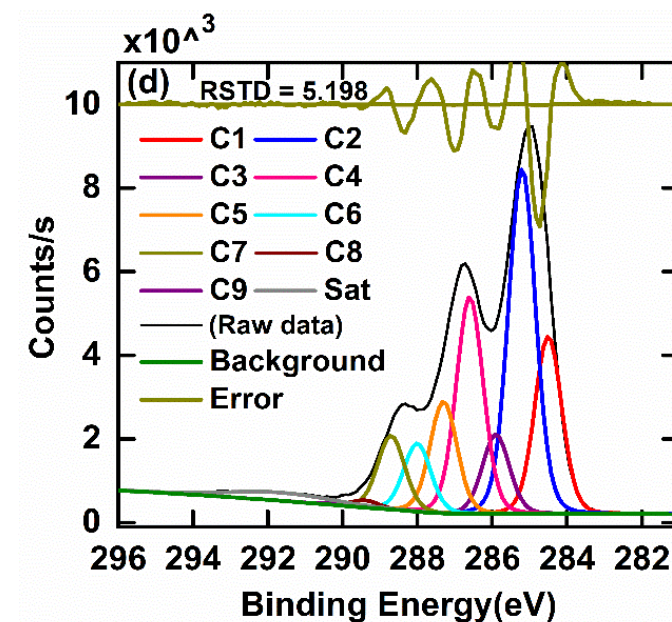
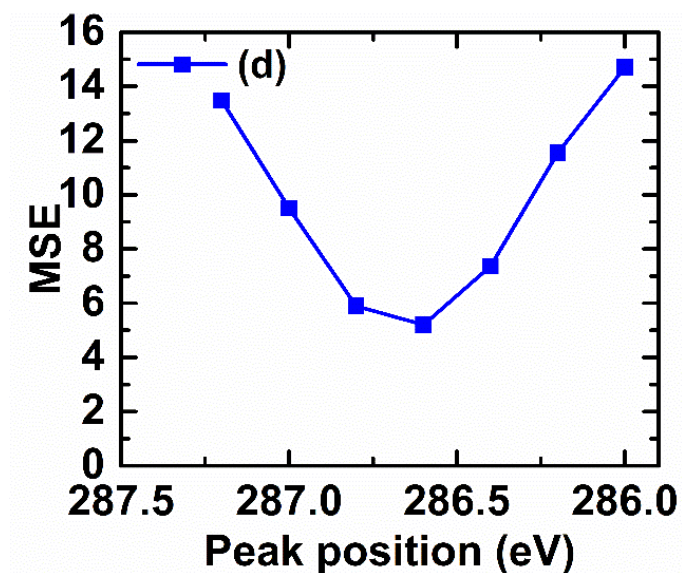
Uniqueness Plots

- Uniqueness plot of the 10-component fit
- *The peaks were fixed at their initial positions, and the nine peaks describing the main peak envelope were constrained to have the same widths*
- Moved the 'C4' component here
- Unique fit



Uniqueness Plots

- Q. Does a bad uniqueness plot mean you have a bad fit?
- A. Probably. At the least, it calls it into question.
- Q. Does a good uniqueness plot prove you have a good fit?
- A. Not necessarily. It really only means you don't have fit parameter correlation.
- It is easier to know what is wrong than what is right in XPS peak fitting.



Outline

Five Projects

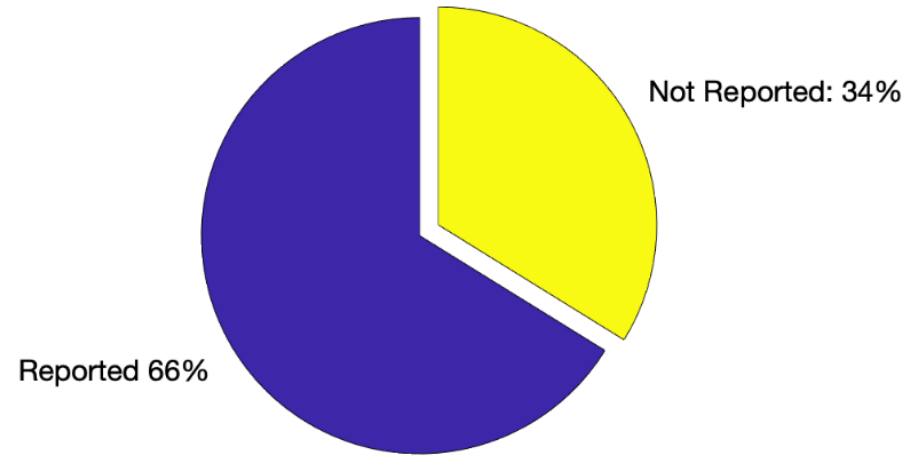
1. Atomic layer deposition in long capillaries
2. Quantifying the number of silanols on fused silica surfaces
3. Quantifying the errors in XPS peak fitting in the literature
4. Uniqueness plots in XPS
5. Lack of experimental reporting in XPS

A reporting study: how we got our data

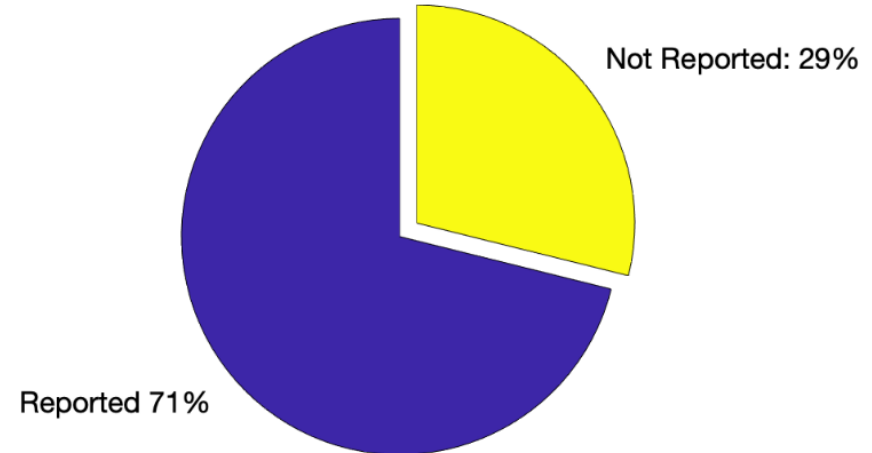
- A reporting study
- What are people reporting or not reporting about XPS?
- The information for this study was obtained from papers published in three high-quality journals over a six-month period in 2019 and throughout 2021.
- Ca. 400 and 850 papers in each year

Lack of Reporting about the Spectrometer

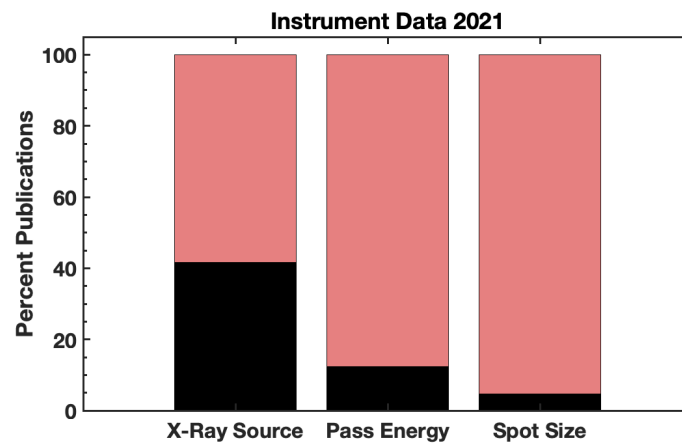
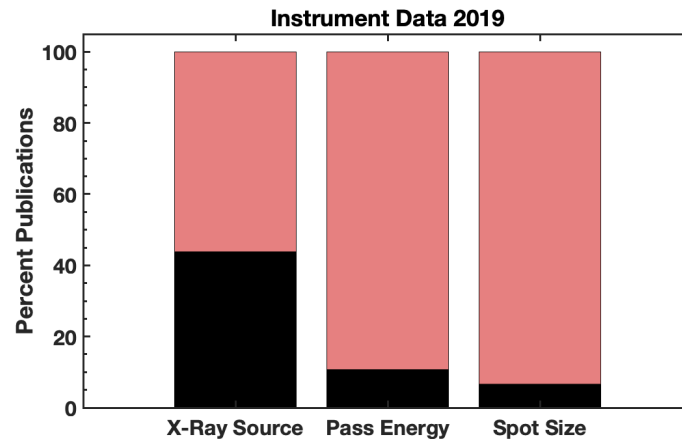
Type of Spectrometer Reported 2019



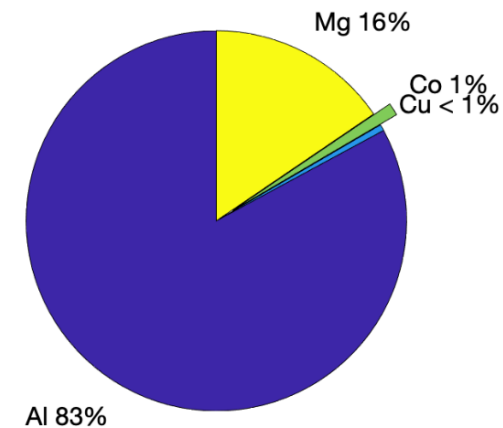
Type of Spectrometer Reported 2021



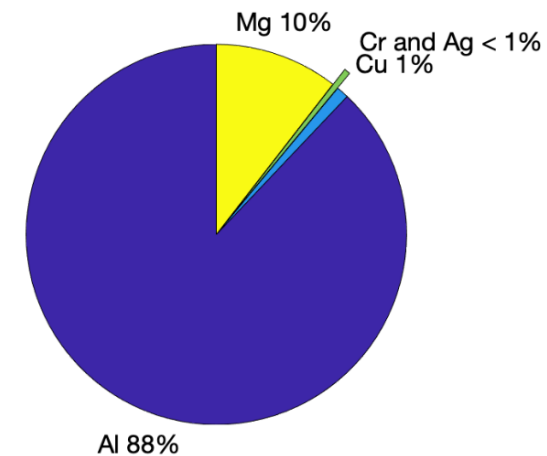
Lack of Reporting about the X-ray Source, Pass Energy, and Spot Size



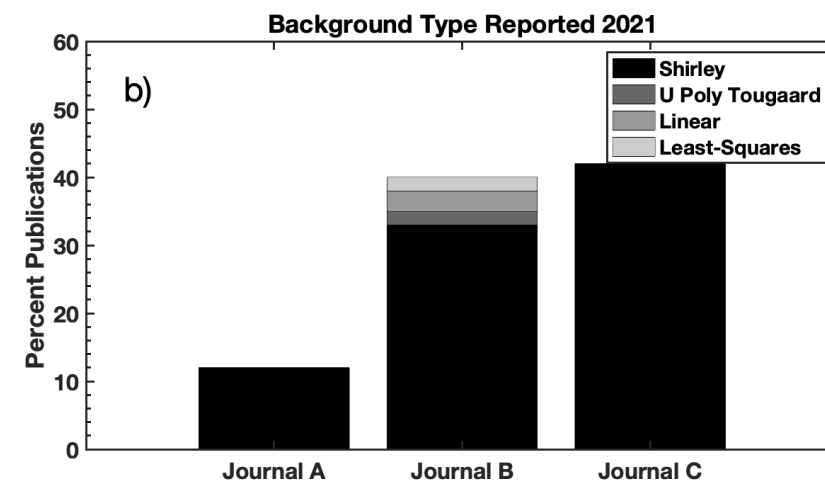
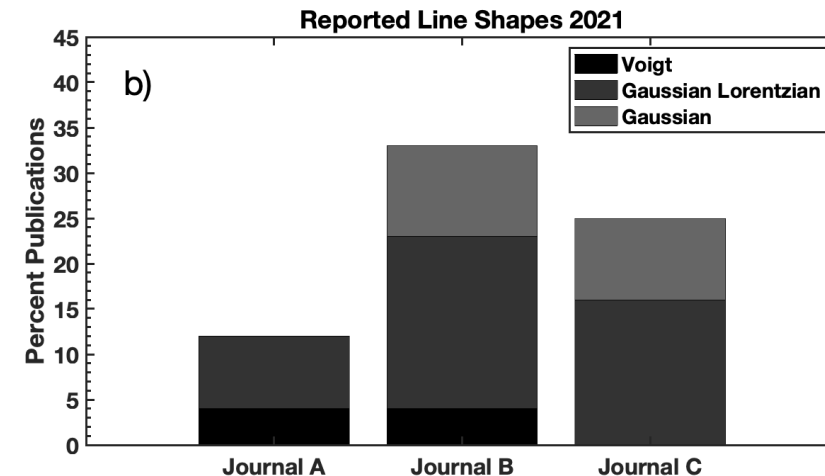
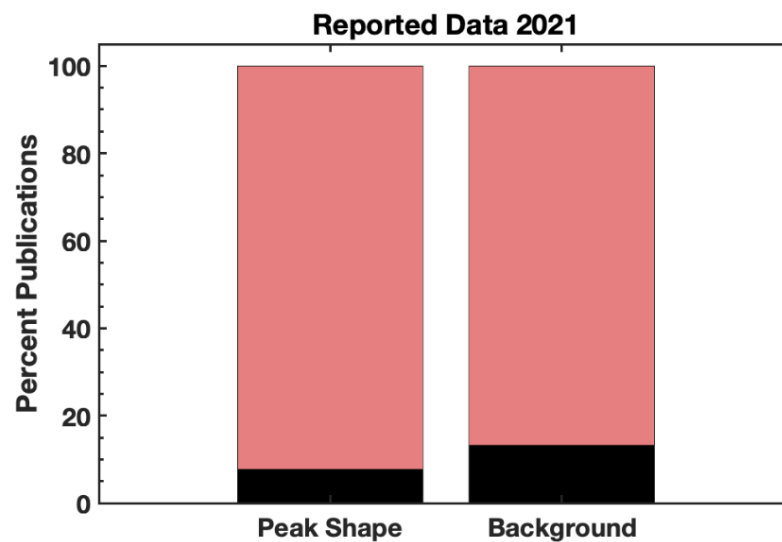
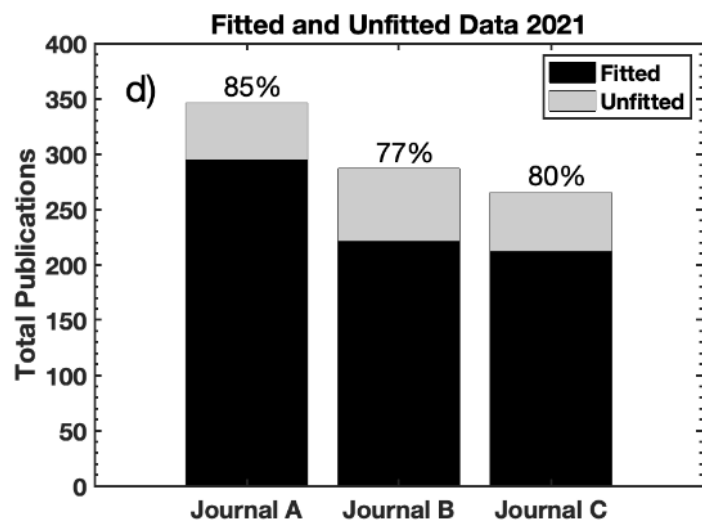
X-ray Source Reported 2019



X-ray Source Reported 2021



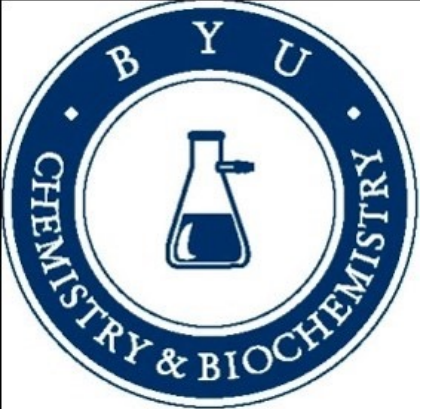
Peak Fitting in 2021



Conclusions

Five Projects

1. Atomic layer deposition in long capillaries
2. Quantifying the number of silanols on fused silica surfaces
3. Quantifying the errors in XPS peak fitting in the literature
4. Uniqueness plots in XPS
5. Lack of experimental reporting in XPS



Acknowledgements



Graduate Student Group members:

Dr. Tuhin Roychowdhury
Dr. Dhruv Shah
Dr. Dhananjay Patel
Dr. Tahereh Avval
George Major
Behnam Moeini
Joshua Pinder
Alvaro Lizarbe
Samira Jafari

Undergraduate Students:

Kevin Cayabyab
Nathan Engel
Max Clark
Jacob Crossman
Annika Dean
Braxton Kulbacki
Steven Ko
Emily Schmidt

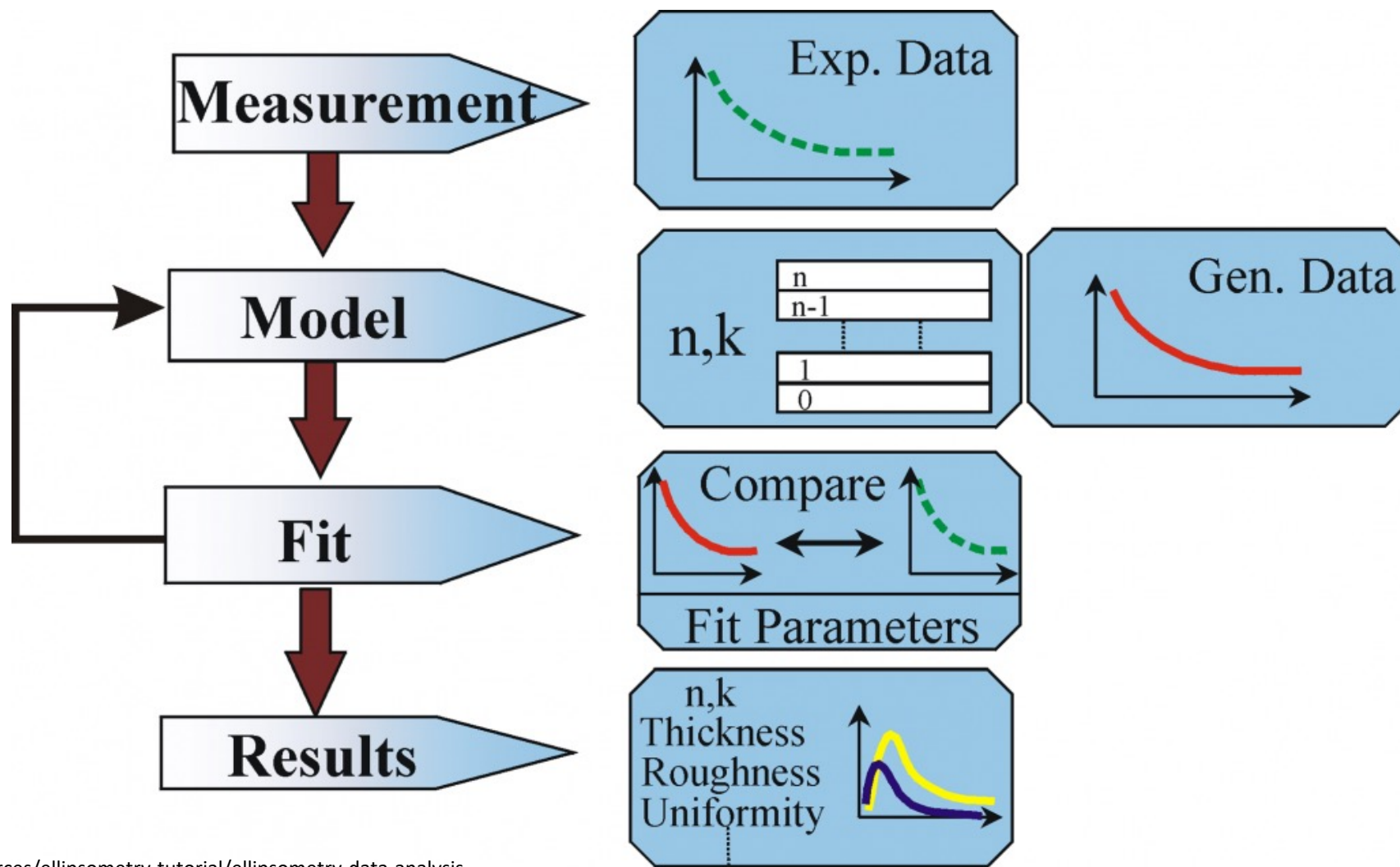
Collaborators:

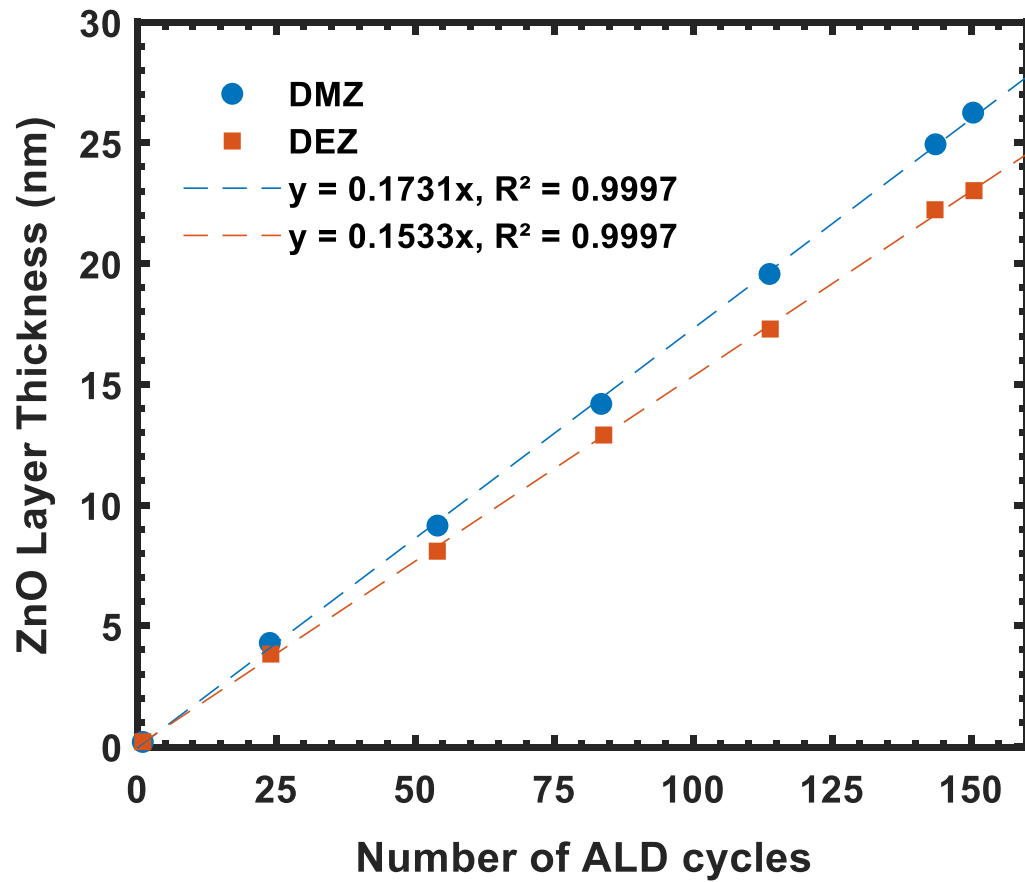
Dr. Don Baer (PNNL)
Dr. Neal Fairley (CasaXPS)
Dr. Vincent Fernandez (U. Nantes)
Dr. William Skinner (U. South Australia)
Dr. Thomas R. Gengenbach (CSIRO)
Dr. Christopher D. Easton (CSIRO)
Dr. Alberto Herrera-Gomez (Cinvestav)
Dr. James N. Hilfiker (JA Woollam)
Dr. Nicholas Smith (Corning)
Dr. Cody Cushman (Corning)
Dr. Stanislav Prusa (CEITEC)



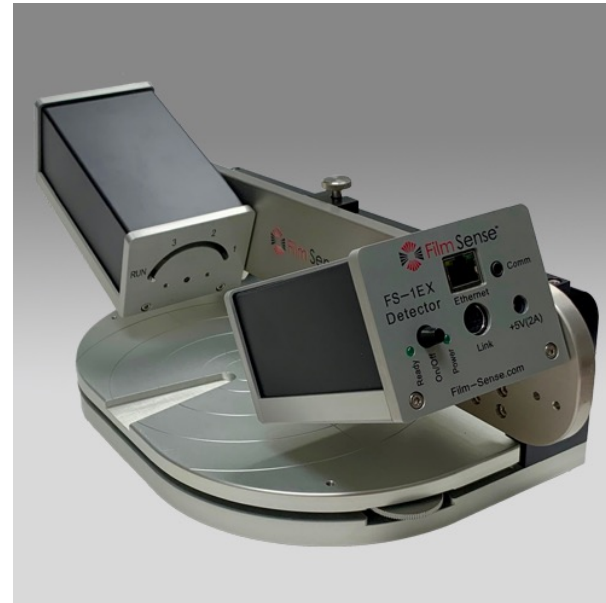
Acknowledgments

- Funders
 - Restek Incorporated, Corning Incorporated, Department of Chemistry and Biochemistry BYU, College of Physical and Mathematical Sciences BYU
- Students and collaborators
 - Listed as co-authors on these projects throughout the talk





- Difference in thickness attributed to less steric hindrance of DMZ



Practical guide for curve fitting in x-ray photoelectron spectroscopy

Cite as: J. Vac. Sci. Technol. A 38, 061203 (2020); doi: 10.1116/6.0000377

Submitted: 5 June 2020 · Accepted: 18 September 2020 ·

Published Online: 6 October 2020



George H. Major,¹ Neal Fairley,² Peter M. A. Sherwood,³ Matthew R. Linford,¹ Jeff Terry,⁴ Vincent Fernandez,⁵ and Kateryna Artyushkova^{6,a)}

AFFILIATIONS

¹Department of Chemistry and Biochemistry, Brigham Young University, C100 BNSN, Provo, Utah 84602

²Casa Software Ltd., Bay House, Teignmouth, United Kingdom

³Department of Chemistry, University of Washington, Seattle, Washington 98195

⁴Illinois Institute of Technology, 3101 S. Dearborn St., Chicago, Illinois 60616

⁵CNRS, Institut des Matériaux Jean Rouxel, IMN, Université de Nantes, F-44000 Nantes, France

⁶Physical Electronics, 18725 Lake Drive East, Chanhassen, Minnesota 55317

Note: This paper is part of the Special Topic Collection on Reproducibility Challenges and Solutions.

a)Electronic mail: kartyushkova@phi.com

ABSTRACT

The use of peak fitting to extract information from x-ray photoelectron spectroscopy (XPS) data is of growing use and importance. Due to increased instrument accessibility and reliability, the use of XPS instrumentation has significantly increased around the world. However, the increased use has not been matched by the expertise of the new users, and the erroneous application of curve fitting has contributed to ambiguity and confusion in parts of the literature. This guide discusses the physics and chemistry involved in generating XPS spectra, describes good practices for peak fitting, and provides examples of appropriate use along with tools for avoiding mistakes.

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I. INTRODUCTION

Over the past 30 years, x-ray photoelectron spectroscopy (XPS) has become the most widely used surface analysis tool and has been an essential component of many research studies.¹ Curve fitting has been widely used for more than 50 years for extracting chemical information from the overlapping features in high-resolution XPS spectra.² Despite computational advances and higher accessibility of software resources, it has been challenging to develop a chemically and physically meaningful approach to curve fitting. The absence of a distinct theoretical description of XPS fitting has led to the publication of erroneous conclusions about surface chemistry.^{2,3} In an ongoing study of XPS data in three high profile journals,³ it was observed that roughly 70% of the papers using XPS analyzed the data using some type of curve fitting. Furthermore, errors, misconceptions, and bad curve-fitting practices accounted for most of the serious problems in both the measured XPS data and the spectral analysis that were identified in more than 30% of the papers analyzed. This guide is intended to help address this important problem.

Curve fitting, also known as peak fitting,⁴ is the process used to extract information from the spectral data for a number of techniques. Although the details of curve fitting depend on the technique in question, the curve fitted spectra generally contain overlapping peaks. Each of these peaks is represented by a function that reflects the physical process involved in generating the original signal. XPS data interpretation and representation range from a rudimentary understanding/extraction of the elements present in a material to advanced peak fitting and background analysis that reveal chemical states and sample morphologies.

In XPS, it is convenient to identify two spectral regions, namely, the core region (electrons with binding energies, BEs, greater than 30 eV) and the valence band region (BE < 30 eV). In the core region, the spectral features arise from photoelectrons generated from core energy levels (atomic orbitals), which are characteristic of the individual atoms in the sample.⁵ In contrast, the features in the valence band region arise from photoelectrons generated from energy levels that typically involve the chemical

Figure(s) from paper George sent

Width Functions

