JVST A
Journal of Vacuum Science & Technology A

Sample handling, preparation and mounting for XPS and other surface analytical techniques

Cite as: J. Vac. Sci. Technol. A **38**, 063202 (2020); https://doi.org/10.1116/6.0000421
Submitted: 30 June 2020 . Accepted: 20 August 2020 . Published Online: 11 September 2020

Fred A. Stevie, Roberto Garcia, Jeffrey Shallenberger, John G. Newman, and Carrie L. Donley

COLLECTIONS

Paper published as part of the special topic on Special Topic Collection: Reproducibility Challenges and Solutions

View Online

Export Citation

CrossMark

ARTICLES YOU MAY BE INTERESTED IN

Practical guides for x-ray photoelectron spectroscopy: Analysis of polymers
Journal of Vacuum Science & Technology A **38**, 023207 (2020); https://doi.org/10.1116/1.5140587

Assessment of the frequency and nature of erroneous x-ray photoelectron spectroscopy analyses in the scientific literature
Journal of Vacuum Science & Technology A **38**, 061204 (2020); https://doi.org/10.1116/6.0000685

Introductory guide to backgrounds in XPS spectra and their impact on determining peak intensities
Journal of Vacuum Science & Technology A **38**, 063203 (2020); https://doi.org/10.1116/6.0000359

Advance your science and career as a member of AVS
LEARN MORE

© 2020 Author(s).

JVST A
Journal of Vacuum Science & Technology A
ARTICLE
avs.scitation.org/journal/jva

Sample handling, preparation and mounting for XPS and other surface analytical techniques

Cite as: J. Vac. Sci. Technol. A **38**, 063202 (2020); doi: 10.1116/6.0000421
Submitted: 30 June 2020 · Accepted: 20 August 2020 ·
Published Online: 11 September 2020

View Online

Export Citation

CrossMark

Fred A. Stevie,[1,a)] Roberto Garcia,[1] Jeffrey Shallenberger,[2] John G. Newman,[3] and Carrie L. Donley[4]

AFFILIATIONS

[1]Analytical Instrumentation Facility, North Carolina State University, Raleigh, North Carolina 27695
[2]Materials Characterization Laboratory, The Pennsylvania State University, University Park, Pennsylvania 16802
[3]Physical Electronics, Chanhassen, Minnesota 55317
[4]Chapel Hill Analytical and Nanofabrication Laboratory, University of North Carolina, Chapel Hill, North Carolina 27599

Note: This paper is part of the Special Topic Collection on Reproducibility Challenges and Solutions.
[a)]**Electronic mail:** fred_stevie@ncsu.edu

ABSTRACT

Sample preparation and mounting are important aspects of x-ray photoelectron spectroscopy (XPS) analysis. New users do not know many techniques that are familiar to analysts with years of experience, and these observations and "tricks of the trade" are typically not published. This article is intended to convey the experience of the authors in this field who have creatively analyzed a wide range of samples. Samples can include solids, powders, fibers, porous solids, and even liquids. This information is also important to anyone submitting samples for analysis, since the preparation of the sample may influence the usefulness of the data collected. These techniques are also applicable to other surface analysis methods.

Published under license by AVS. https://doi.org/10.1116/6.0000421

I. INTRODUCTION

This article is part of a series of papers intended to provide newer users with a better understanding of x-ray photoelectron spectroscopy (XPS), the ability to obtain quality data and perform appropriate data analysis.[1] Recent articles have pointed out quality and reproducibility issues with many types of material data analysis in the literature.[2,3] The goal of this series is to help educate the scientific community that utilizes XPS, and sample handling and preparation are the necessary first steps to collecting quality data.

X-ray photoelectron spectroscopy is the most widely used surface analytical technique, and it probes the top ∼10 nm of the surface of the sample. This surface sensitivity requires the user to carefully protect the region of interest, which can easily be compromised. Contamination due to handling, sample storage, and exposure to the atmosphere can affect the results. A number of books provide sections on sample preparation but often do not provide many details.[4–7] In addition, guides for sample preparation and mounting have been provided by ASTM and the International Standards Organization (ISO).[8–12] This paper draws on the experience of a number of analysts who have to solve sample issues every day and presents their combined knowledge.

Two important aspects of sample preparation are considered in this paper: (i) sample handling and preparation before analysis and (ii) sample mounting for the XPS measurement. Preparation may involve alteration of the as submitted sample so that it can be mounted on an available sample holder, while mounting is concerned with the attachment of the sample to the holder.

II. SAMPLE HANDLING AND PREPARATION

XPS analysis occurs in vacuum, and, therefore, this paper is primarily devoted to the analysis of solid samples, but a few suggestions for liquids are also offered. Many samples can be analyzed as received, and others only require the sample to be cut so that it will fit on the sample holder. The allowed sample size depends on the instrument configuration, but commonly samples on the order of $\sim 1 \times 1\ \mathrm{cm}^2$ and less than 1 cm high can be analyzed in most systems. Some instruments can accommodate samples as large as a 300 mm diameter silicon wafer.

Since the XPS experiment is very surface sensitive that the surface must be protected from contamination, unwanted surface oxidation, etc., and it is essential to communicate this to anyone who plans to submit a sample for analysis. Samples should be

Published under license by AVS.

stored in containers such as Fluoroware or glass, or wrapped in aluminum foil, or folded in a copier paper. Plastic bags should be avoided as they often contain silicone such as polydimethylsiloxane (PDMS) or slip agents such as erucamide, which can be transferred to the sample surface. Other potential contaminant sources include the lids on glass jars, desiccators with vacuum grease seals, plastic syringes, and containers with elastomers.

The region of the surface to be analyzed should not be touched, even with gloves or tweezers. A single fingerprint can completely cover the region of interest and can even affect the base instrument vacuum level,[13] and gloves can also be a source of contamination if the gloves touch the surface to be analyzed.[14] A typical procedure is to wear powder-free, silicone-free latex or nitrile gloves and manipulate the sample by its edges with cleaned stainless steel tweezers. Stainless steel tools can be easily cleaned with solvents such as methanol.

To reduce the sample to the desired size, cutting with sharp laboratory quality scissors is advised. Ordinary desk scissors can cause a thin sample to fold on itself when cut, and laboratory quality scissors can be obtained in various sizes and can cut stainless steel at least 0.4 mm thick. Serrated blade scissors are handy for thin foils, and thicker samples may require cutting with shears. Other tools for cutting include a diamond saw lubricated with water (de-ionized is preferred) and not oil or simple tools such as hacksaws. If cutting will produce particles, the region of interest must be protected, and taping a piece of paper over this region is recommended.

In general, semiconductor samples can be cleaved by nicking the edge with a diamond scribe. Silicon, GaAs, and InP can easily be cleaved in this manner, but SiC, sapphire, and GaN are much harder, and cleave lines may be nonrectilinear, similar to Si (111). Glass samples can also be scribed and fractured as described above or fractured with specially designed tools. Two examples are a pliers with curved ends, called running pliers, which fracture the sample in the middle of the curved end, and a pliers with sharp wheels for scoring the glass, as shown in Fig. 1. Often particles are generated as a result of fracture methods, and they can be removed with a spray nozzle hooked up to a source of compressed gas. A gas cylinder of air or dry nitrogen is recommended to blow off the sample surface. Compressed air from a central location typically relies on a compressor which is lubricated with oil that can contaminate the sample. Hand-held "canned air" spray cans designed to clean out keyboards are not advisable as they do not actually spray air, but typically fluorinated hydrocarbons, and may also contain a bitterant, which will contaminate the sample.

Cleaning the surface is not advised, but sometimes cannot be avoided. For chemical cleaning, hexanes can help remove some common lubricants, such as PDMS, and probably provide the least disturbance to the surface. The final rinse should be done with isopropyl alcohol (IPA) or methanol as these leave the least amount of organic residue on the sample. Acetone should not be used. It is suggested to rinse the sample with the solvent in a hood and then immediately blow dry with compressed gas. One should avoid wiping the surface with a cloth.

If adventitious carbon or other surface contamination is limiting the detection of the species of interest, the surface can be sputter cleaned within the XPS vacuum chamber. Many older instruments have only an argon ion beam available for sputtering. This must be done with care as sputtering with an Ar^+ beam can damage the surface; it is known to reduce some metal oxides (TiO_2) and can seriously damage polymers. It is recommended that the energy of the ion beam be kept as low as possible to minimize sample damage. Typically, sputtering with an Ar^+ beam is conducted on pure metal surfaces, such as Au, Ag, or Cu (common reference materials for XPS), to produce clean surfaces in vacuum. In general, the sputtering process will modify the surface to the depth of penetration of the ion beam and can amorphize crystalline materials. For example, a 5 keV Ar^+ beam will penetrate silicon to a depth of 13 nm (projected range plus straggle calculated with TRIM),[15] which exceeds 10 nm normally considered as the analysis depth in XPS.

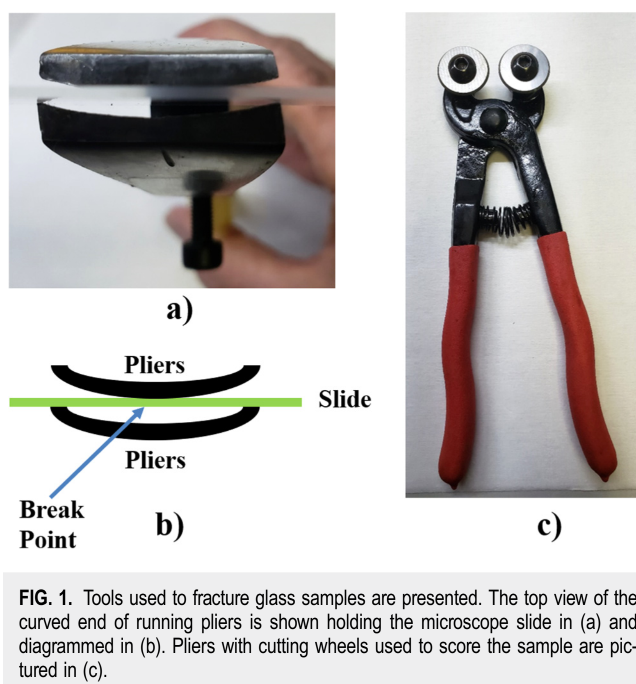

FIG. 1. Tools used to fracture glass samples are presented. The top view of the curved end of running pliers is shown holding the microscope slide in (a) and diagrammed in (b). Pliers with cutting wheels used to score the sample are pictured in (c).

Sputtering sources can also be used to provide an XPS depth profile,[16] and while this has been done with Ar^+ sources, newer sources, such as C_{60} or a gas cluster ion beam (GCIB), can provide depth profiles in many organic materials without affecting the chemical state of the material.[17–20] These GCIB and C_{60} sources can also remove adventitious carbon.

While sputtering is one way to measure the composition at depths greater than 10 nm, another method is to reduce the sample to a powder. Powder samples are often analyzed with XPS and a discussion of mounting options for powders is provided in Sec. III.

Some samples that are porous or polymer based will tend to incorporate volatile materials, and this will make it difficult to achieve a low enough pressure for analysis. It is recommended that the volatiles are removed by extended pumping in a sample introduction chamber or other vacuum station prior to analysis.

Samples sensitive to air exposure require special handling to prevent unwanted surface oxidation reactions from occurring

Published under license by AVS.

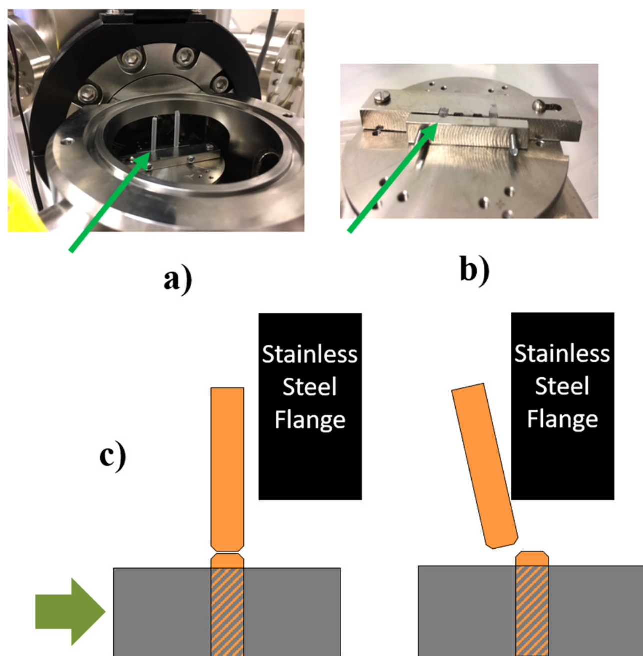

FIG. 2. (a) Sample fracture method is shown where samples break on a steel flange and (b) the postfracture view. The arrows show the sample before and after fracture. (c) shows a schematic drawing of the method.

during the sample loading process. Most vendors now offer some type of sample fracture system to cleave the sample in vacuum to expose a fresh surface not exposed to ambient conditions. Figure 2 shows a method of generating a freshly fractured surface in the vacuum environment of the sample introduction chamber. The sample is mounted in the vacuum chamber so that it is intentionally too tall to enter the analysis chamber. The sample chamber is pumped to the desired vacuum, and the sample is fractured by moving the sample across the edge of a stainless steel flange.

In addition, many vendors also offer some type of inert atmosphere transfer vessel, which can be operated under vacuum or inert atmosphere. These are commonly small enough that they can fit into the entry port of a glovebox for sample loading. The sealed container containing the samples is used to transport the samples to the XPS, where a mechanism exists to attach this transfer chamber directly to the XPS sample introduction port to load and loads the samples without exposure to air. This is of particular interest for the analysis of battery materials that contain lithium and magnesium or air sensitive catalysts. An example of a commercial transfer vessel is shown in Fig. 3. These chambers may not be available to all users, and creative, low cost solutions have also been implemented. A common approach is to load the samples onto XPS sample holders in a glovebox and then enclose and seal the samples in an argon or nitrogen filled container (plastic bag, sealed glass container, etc.). The samples are transferred to the instrument and the transfer container is placed within a glove bag attached to the XPS sample loading port. The glove bag is purged with nitrogen prior to venting the XPS chamber to nitrogen and opening the transfer container to load samples. Attachment of a glovebox directly to the XPS sample chamber has also been reported.[21]

Because of the vacuum requirements for XPS, liquid samples are difficult to analyze and often require specialized equipment not available in all laboratories. One approach is to freeze the liquid by maintaining a cryogenic temperature during pump down and during analysis.[22] Biological and other wet samples require special treatment during pump down in order to avoid sample damage, and cryogenic analysis has proven to be a successful approach.[23,24] Cryogenic systems cooled with liquid nitrogen allow for sample

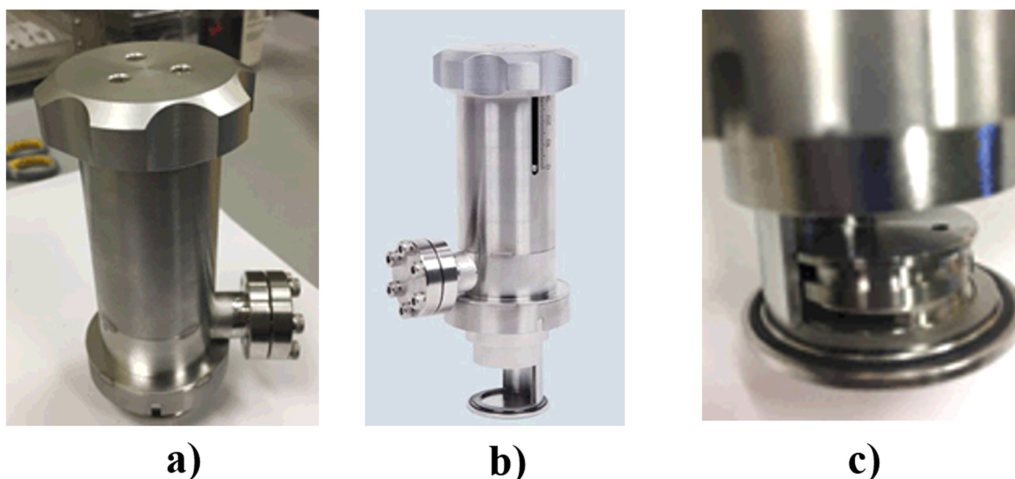

FIG. 3. Sample transfer vessel for Physical Electronics instruments is displayed: (a) a closed vessel, (b) a vessel open to the load sample, and (c) a vessel with a sample holder. Samples can be transferred in vacuum or inert gas directly into the sample introduction port of the XPS.

Published under license by AVS.

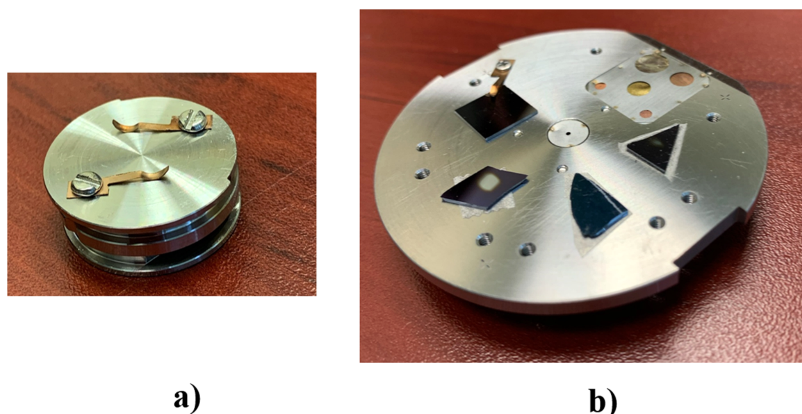

FIG. 4. (a) 25 mm diameter and (b) 60 mm diameter flat sample holders shown for Physical Electronics instruments.

temperatures below −100 °C. Near ambient pressure XPS (NAP-XPS) instruments have been used to analyze biological samples and liquids or solids with high vapor pressure.[25,26] Analysis with pressures on the order of 25 mbar (2500 Pa) has been achieved using differential pumping.

Many analytical methods may either modify the surface chemistry of the sample or contaminate the surface, and XPS is generally performed before using other analytical techniques. For example, electron beam techniques such as SEM are known to deposit carbon on the sample surface due to interactions between the electron beam and residual pump oils in the vacuum chamber. An exception is static SIMS operated under true static limit conditions in which only ~0.1% of the sample surface will be damaged during the analysis. This is commonly achieved with a time of flight instrument (ToF-SIMS).

Although XPS is often considered a nondestructive technique, it too can cause damage to sensitive samples. Sometimes, samples will show visible discoloration where the x-ray beam was incident. Paper or PVC samples can show spots from degradation, alumina shows spots from x-ray induced color-centers, and some organics photoreduce. If visible sample degradation occurs, care should be taken to avoid analyzing this region with other techniques, and the careful analyst will investigate XPS induced changes in the sample that could influence the interpretation of the XPS data. Cooling the sample with liquid nitrogen during the analysis can sometimes minimize these damage effects.

Radioactive materials should not be placed on instruments. Any possibility of contamination will require special and expensive procedures if servicing is required. Components such as pumps cannot be exchanged and must be treated as radioactive waste. There are groups that specialize in the analysis of radioactive materials, and they have instruments dedicated to this purpose.[27]

III. SAMPLE MOUNTING

A. General considerations

XPS can detect submonolayer coverages, so proper sample handling is essential. As mentioned above, gloves should be worn and clean metal tweezers used to handle the sample ***without contacting the area of interest***. Samples should be stored and transported in a way that does not introduce surface contamination.

A sample size of $1 \times 1\ \text{cm}^2$ and less than 1 cm high $1 \times 1 \times <1\ \text{cm}^3$ can be analyzed in most instruments, but the actual allowed sample size is instrument dependent. Samples with lateral dimensions less than a few mm are more difficult to mount and more care is required to avoid analyzing parts of the sample holder. Examples of sample holders are shown in Figs. 4–7 from several vendors. Many sample holders hold specimens with metal clips or masks directly attached to the holder. Some of the sample holders shown have the ability to be rotated during a depth profile analysis, specifically

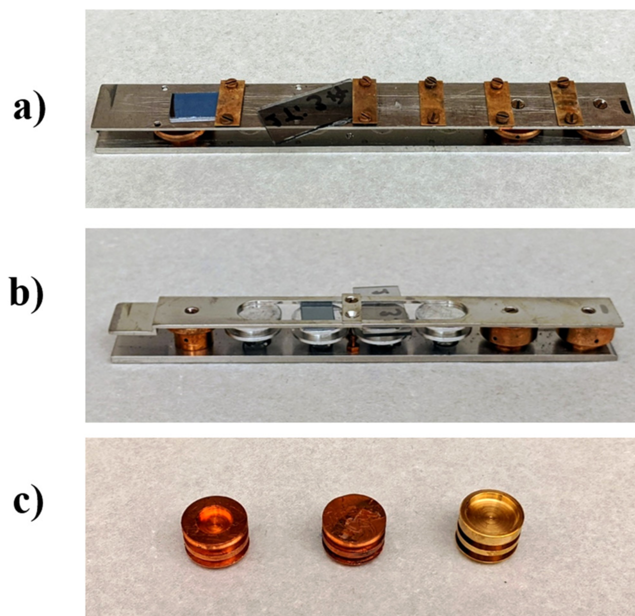

FIG. 5. Sample holders are shown for the Kratos Axis Ultra-DLD instrument: (a) the standard multisample holder, (b) the spring loaded constant height sample holder, and (c) holders for small samples, powders, and irregular shapes.

Published under license by AVS.

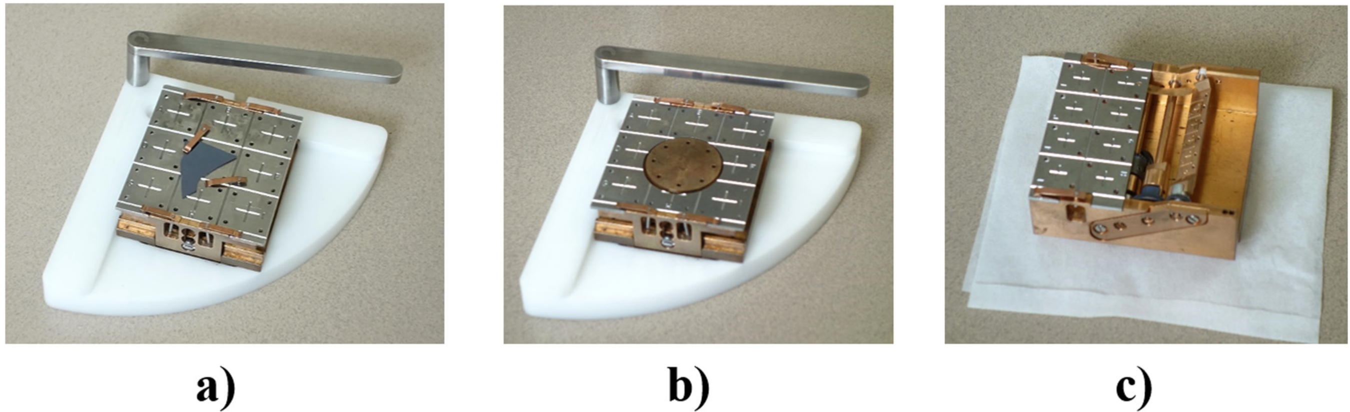

FIG. 6. Sample holders are shown for the ThermoFisher K-alpha instrument: (a) a standard sample holder (the swing arm is used to check sample height), (b) a rotating sample holder, and (c) a tilt sample holder.

those in Figs. 4, 6(c), and 9. Rotation of the sample during sputtering can produce a more uniform etch crater during a depth profile, especially when using monatomic argon ion guns.[28,29]

XPS is used to analyze such a wide variety of samples that a number of approaches have been developed for sample mounting.[30,31] Silicone-free double-sided conductive sticky carbon tabs or tape can be used to attach the sample to the holder. If small pieces are used and prepumped in an entry chamber, then operating pressures in 10^{-10} mbar can be maintained in the analysis chamber. Semiconductor samples are often fragile, and it can be difficult to remove them from the sample holder if the tape is too sticky. In these cases, a less sticky type of tape can be used. Sample holders with metal clips are also common as shown in Figs. 4, 5(a), 6(a), 7(b), and 7(c). Often metal clips are a preferred method of securing

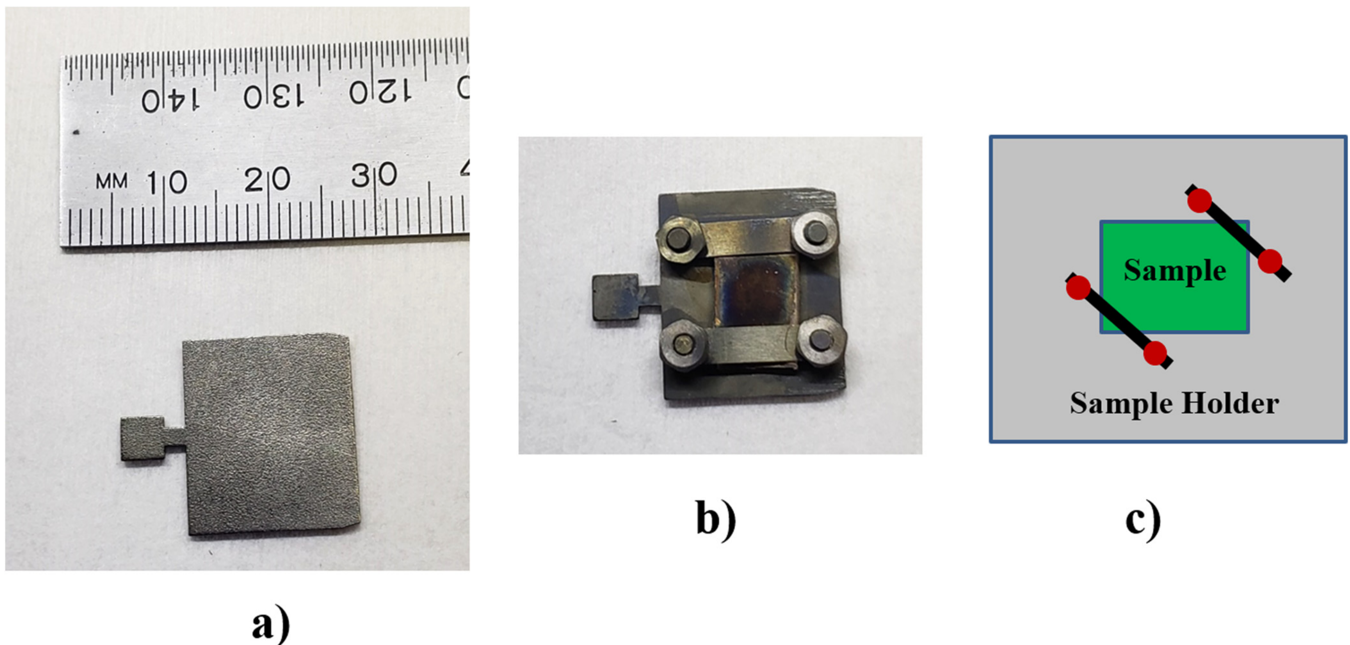

FIG. 7. Sample holders are displayed for the SPECS instrument: (a) a plain holder, (b) holder with metal strips attached to posts, (c) diagram of spot-welded strips of metal to retain the sample. Holder types (b) and (c) are useful if the sample is to be heated or cooled.

Published under license by AVS.

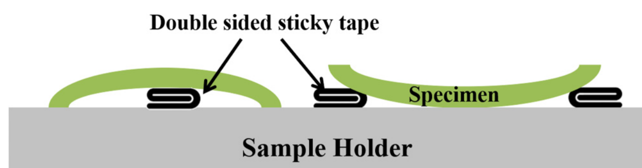

FIG. 8. Mounting of nonflat specimens is shown. A double-sided sticky tape folded over several times can be used to fill the gap between the specimen and sample holder for concave and convex sample shapes.

a sample because they will make better electrical contact to the surface of the sample than adhering the sample from the bottom with double-sided tape. Samples that need to be heated or cooled cannot be mounted with tape as the tape greatly reduces temperature transfer and can heavily outgas at elevated temperature, and thus mechanical attachment is required.

B. Irregular shapes

Samples for XPS do not need to be smooth and flat. Samples with curved surfaces, either concave or convex, and spherical shapes, such as beads and balls, can be analyzed. For example, the inside and outside surfaces of tubing can be analyzed by cutting the tubing into half. This is easily accomplished with plastic tubing but cutting metal tubing can be more difficult and poses a risk of sample contamination. Figure 8 shows a method to hold concave or convex shapes. Samples that are difficult to hold with a clip can be placed in a recessed sample holder as shown in Fig. 5(c) and Fig. 9.

Some instruments use rear mounted sample holders such as the spring loaded sample holder as shown in Fig. 5(b). This approach keeps all samples top referenced on the holder so that they are at the same distance to the analyzer. Metal "stands" can also be used to mount thin samples next to thick samples so that the sample surfaces are at similar heights. This avoids the possibility of hitting the analyzer optics with a tall sample when analyzing a nearby thin sample.

Pretilted mounts can be used for special geometries as shown in Fig. 6(c) and Fig. 10. These types of holders can also be used for angle dependent measurements if it is not possible to tilt the sample holder on the instrument. A sample holder designed to hold multiple samples for angle dependent XPS measurements is shown in Fig. 11. When the cross section of a sample is required, a mini-vise, as shown in Fig. 12, can be used to mount the sample in the desired orientation.

C. Powders

Many types of powders are commonly analyzed using XPS and include building materials, soil samples, burnt wood, nanomaterials, catalysts, carbon black, and particles used in 3D printers.[19,30,32] The powder can usually be removed from a storage bottle with a small spatula and then spread over a double-sided tape that has been placed on the sample holder. The powder can then be pressed into the tape. A permanent tape is more sticky than other versions and makes it ideal for powder materials. Another successful method is to press the powder into a soft material, usually indium, with a clean spatula. Exposing a fresh indium metal surface by scraping the oxide off with a thin razor blade is recommended to provide better electrical contact. For both methods, it is important to remove loose particles by tilting the sample holder and tapping it gently on a surface. You do not want loose particles to get into the extraction column, onto a gate valve gasket, or get through the protective shield on a turbomolecular pump and cause damage. Ideally, a thin layer of powder will

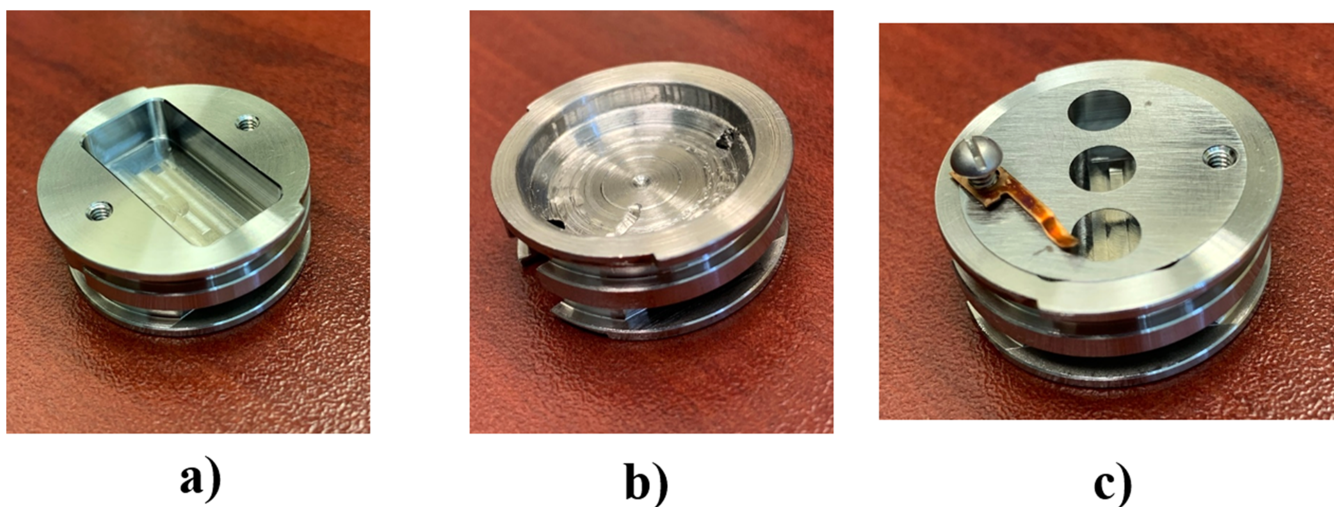

FIG. 9. Recessed sample holders shown with (a) rectangular, (b) circular area, and (c) cover over the recessed area with three analysis openings. Images provided by Physical Electronics.

Published under license by AVS.

FIG. 10. Pretilted sample holders are shown. Images provided by Physical Electronics.

uniformly cover the material used to hold it (tape/indium), but sometimes signal from the substrate will be observed. When working with indium, it is a good idea to include an In 3D high resolution scan. Peak fitting can separate the In^0 and In_2O_3 surface oxidation components, which is important when quantifying oxygen in the sample.

Powders can be pressed into a pellet using a pellet press, such as that used for IR samples, but the pressing surface must be very hard and clean. A similar approach is to pack powders, such as carbon black, into holes drilled into a metal plate. If available, a powder can be loaded into a small metal tray or depression on a holder as long as it does not tip during pump down and analysis. The amount of powder used should be as small as possible, and with this approach, powders can be analyzed at elevated temperatures. (Tape will degrade and outgas with heating, and the melting point of indium is 157 °C.) Figure 13 shows a method of mounting loose powders using a sticky tape and metal washers. The XPS signal is mostly from the first few nanometers so a thin layer of powder is sufficient.

Powders that become charged by static electricity can be difficult to place on the sample holder with a spatula as the powder will fly off in all directions. A method that has been successful is to invert the bottle at an angle so the mouth is just over the sticky tape, tap the bottle with a spatula or tweezers, and slowly pour the powder directly on the tape or indium foil. Antistatic devices may also help reduce this problem.

Another way to work with powders is to disperse or dissolve them in some solvent that they do not react with, and then to drop cast them onto a clean surface such as a silicon wafer or a gold coated silicon wafer. Sometimes, this thin film will show fewer charging effects compared with the original powder mounted in other ways.

D. Semiconductors and insulators

Even though x rays (naturally uncharged) are used as the probing beam in XPS, the photoelectron, Auger, and secondary

FIG. 11. Sample holder designed to hold several samples for angle dependent measurements. Image provided by Physical Electronics.

FIG. 12. Mini-vise can be used to mount samples for cross-sectional analysis. Image provided by Physical Electronics.

Published under license by AVS.

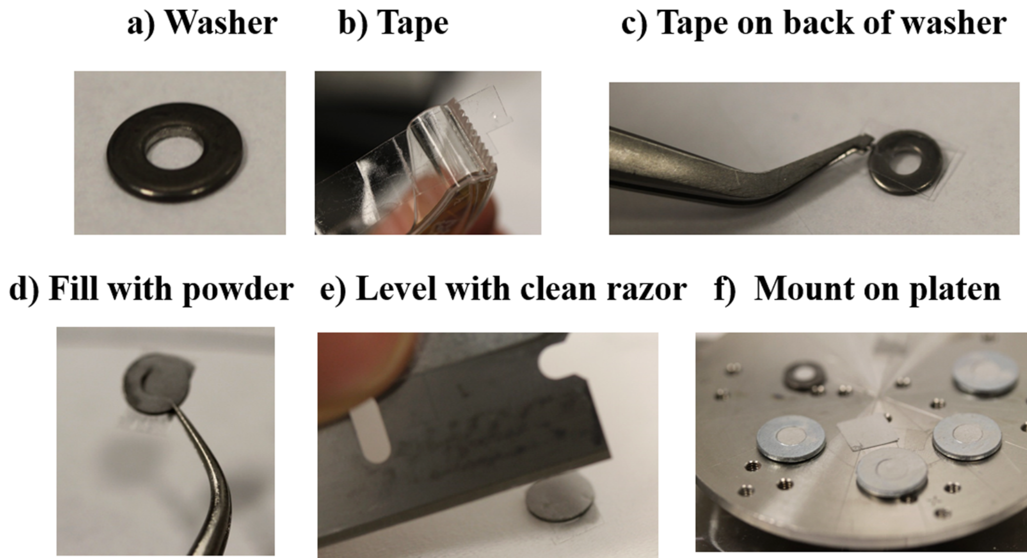

FIG. 13. Mounting method for powders shows the use of washers filled with powder as shown in figures (a)--(f).

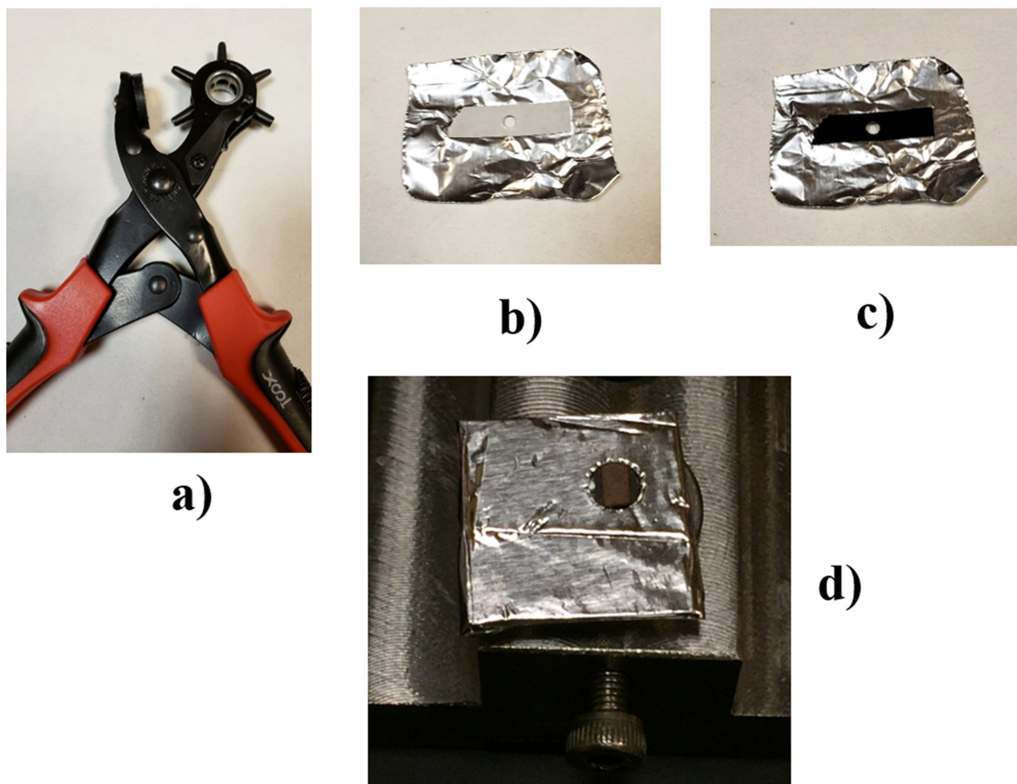

FIG. 14. Aluminum foil method is shown for analysis of insulators. Leather punch (a) is used to make a hole in the aluminum foil and the double-sided carbon tape as shown in (b). Once the other side of the tape cover is removed as shown in (c), aluminum can be placed on the sample over the region of interest as shown in (d). A cover with a hole can also be used as shown in Fig. 9(c).

Published under license by AVS.

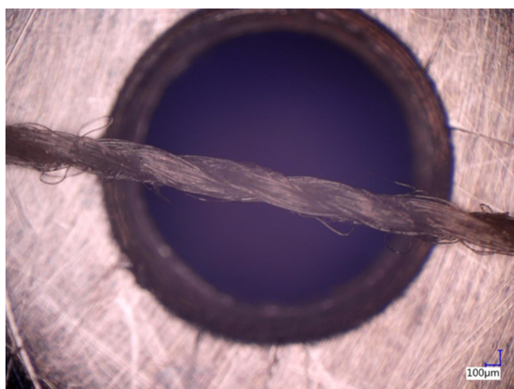

FIG. 15. Fiber shown mounted above a hole in the holder. Image provided by Physical Electronics.

electrons that leave the sample will cause the sample to charge positively. Coating the sample with a conductive gold or gold-palladium layer (as is common in scanning electron microscopy) is not an option because this will prevent XPS analysis of the original surface. For good energy resolution on all types of samples, it is critical to maintain a constant surface potential during the XPS measurements. For semiconducting materials, a constant surface potential can often be obtained by grounding the sample by making electrical contact between the sample surface and the sample mount. This can be done by using metal clips, silicone-free double-sided carbon, copper tape, or silver epoxy to provide a conductive path from the sample holder to the top surface. Placing a mask, consisting of a conductive foil or sheet with a hole in it, on top of the sample is another method of dissipating surface charge. Conductive masks often improve the conduction path to ground compared with attaching a clip or conductive tape at the edge of the sample, and they minimize the possibility of the low energy neutralizing electron or ion beam(s) being deflected prior to reaching the area of analysis. Commercial masks are shown in Figs. 4(b) and 9(c). Cheap homemade masks can be fabricated by cutting an aperture in a piece of aluminum foil with a hole punch, similar to that used to make holes in belts. The mask is typically placed on top of the sample and can be adhered with double-sided tape as long as the area of interest remains exposed. Figure 14 shows the hole punch and aluminum mask with tape and a mounted sample in Fig. 14(d). With high spatial resolution instruments, a 3 mm transmission electron microscope grid can be placed over the sample, and the sample can be analyzed between the lines of the grid.[33]

For insulating materials, a different approach for obtaining a constant surface potential must be used, as grounding the sample is not possible. All modern XPS instruments have some form of charge neutralization system to compensate for the positive charge buildup on insulating samples during x-ray bombardment. Most systems use either a low energy electron flood or a mixture of both low energy electrons and low energy argon ions.

For samples that contain both conducting and insulating domains, it can be beneficial to isolate the sample from the grounded sample mount to avoid what is referred to as differential charging.[30] Differential charging occurs when part of the sample is at one electrical potential (usually grounded) and other parts are at a different potential. This can occur in heterogeneous samples where thin insulating oxide layers, corrosion material, organic residue, surface debris, etc. are present on top of conducting or semiconducting

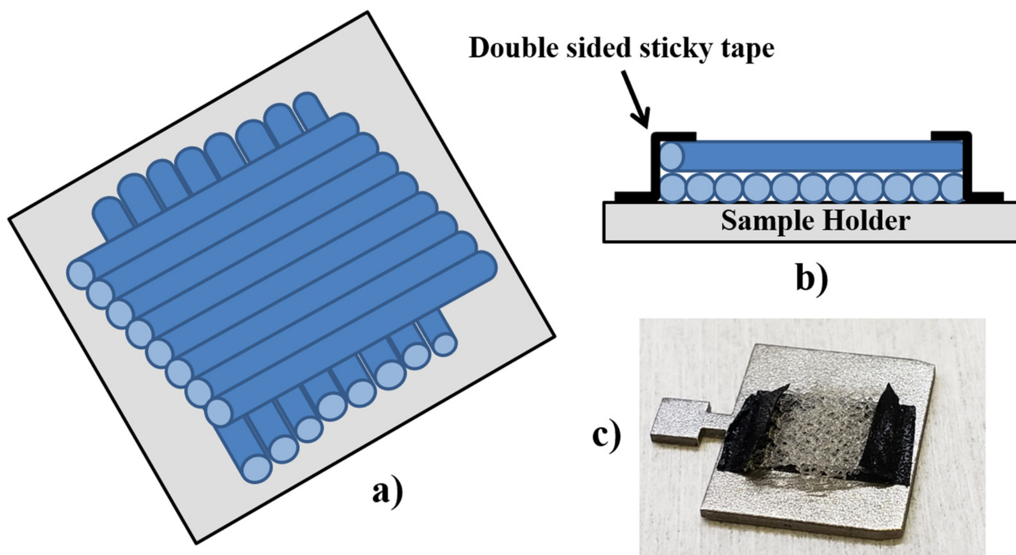

FIG. 16. (a) Two layers of fibers can be arranged to provide a continuous fiber surface for analysis. (b) Tape can be used to retain the assembly on the holder. (c) An example is shown of porous mesh mounted on a holder.

Published under license by AVS.

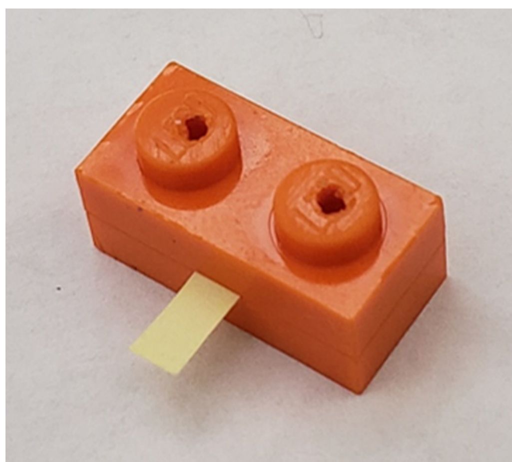

FIG. 17. Fiber and fabric samples can be retained with plastic toy blocks. Holes are drilled through the blocks to facilitate pump down.

substrates. Isolating the sample from electrical ground allows the whole sample to float at a fixed potential dictated by the charge neutralization conditions. Isolating the sample from the mount is easily performed using a nonconductive double-sided tape.

E. Fibers and fabrics

A proven method for mounting fibers is to lay them over a hole in the sample holder and to secure each end with a screw as shown in Fig. 15. The sample over the hole can be analyzed without fear of detecting signals from the sample holder. Another approach is to arrange two layers of packed fibers with the top layer oriented 90° with respect to the bottom layer as shown in Fig. 16(a). A tape can be used to hold down the "mats" as diagrammed in Fig. 16(b). An example of a porous mesh mounted in several layers is provided in Fig. 16(c).

Some plastic toy blocks are made of precisely manufactured hard plastic. Fibers and fabric can be easily mounted with the use of small pieces. Figure 17 shows a small piece of paper clamped between two blocks. Holes have been drilled through the blocks to facilitate pumping.

F. Specific sample types

Concrete, brick, asphalt, and wood tend to be porous and may require a long time to pump down. If possible, this is the type of sample to schedule for analysis after pumping over a weekend. If porous samples can be reduced to a powder, the pump down time should be close to normal. Some systems will have provisions for heating the sample in the sample introduction chamber, which can be useful for samples that outgas significantly.

Some polymers and biological samples often do not adhere well to the tape. In these cases, clips or masks can be used to secure these samples, or the sample can be held by using tape on opposite edges of the sample as was shown in Fig. 16. In addition, some biological samples will often have irregular shapes requiring special mounting procedures. Plant materials and biological tissue may be affected by pumping and may require cryogenic preparation and analysis. Special handling precautions are required if the sample presents a biohazard.

Magnetic fields produced by magnetic samples can affect the trajectory of emitted electrons and can magnetize materials in the instrument and affect instrument performance. Instruments that use a magnetic lens have more problems with magnetic samples, and it may be necessary to operate the instrument in the electrostatic mode (with the magnet off) for these samples. The strength of the magnetic sample should be determined before it is put into an instrument. If a small paper clip is attracted to the sample, the magnetic field of the sample may be too strong to place it in the instrument.

IV. SUMMARY

Sample handling, preparation, and mounting are important aspects of XPS analysis, and surface cleanliness is essential for meaningful results. Methods for sample preparation and mounting have been discussed to aid the analysis of a wide range of materials and material types. The information in this paper is generally applicable to other surface analysis methods such as Auger electron spectroscopy and secondary ion mass spectrometry.

ACKNOWLEDGMENTS

This work was performed, in part, at the Analytical Instrumentation Facility (AIF) and the Chapel Hill Analytical and Nanofabrication Laboratory (CHANL), both of which are supported by the State of North Carolina and the National Science Foundation (Award No. ECCS-1542015). The AIF and CHANL are members of the North Carolina Research Triangle Nanotechnology Network (RTNN), a site in the National Nanotechnology Coordinated Infrastructure (NNCI).

REFERENCES

[1] See: https://avs.scitation.org/toc/jva/collection/10.1116/jva.2020.REPROD2020.issue-1.
[2] D. R. Baer *et al.*, J. Vac. Sci. Technol. A **37**, 031401 (2019).
[3] M. R. Linford *et al.*, Microsc. Microanal. **26**, 1 (2020).
[4] J. F. Moulder, W. F. Stickle, P. E. Sobol, and K. D. Bomben, *Handbook of X-ray Photoelectron Spectroscopy*, edited by J. Chastain (Physical Electronics Division, Perkin-Elmer, Eden Prairie, MN, 1992), pp. 12–14.
[5] J. F. Watts and J. Wolstenholme, *An Introduction to Surface Analysis by XPS and AES* (Wiley, Chichester, 2020), pp. 145–154.
[6] D. Briggs and M. P. Seah, *Practical Surface Analysis, Vol. I. Auger and X-ray Photoelectron Spectroscopy* (Wiley, Chichester, 1990), pp. 36–51.
[7] J. Geller, *Surface Analysis by Auger and X-ray Photoelectron Spectroscopy*, edited by D. Briggs and J. T. Grant (IMPublications, Chichester, 2003), pp. 89–116.
[8] ASTM E1829-14, *Standard Guide for Handling Specimens Prior to Surface Analysis* (ASTM, West Conshohocken, PA, 2014).
[9] ASTM E1028-02, *Standard Guide for Specimen Preparation and Mounting in Surface Analysis* (ASTM, West Conshohocken, PA, 2002).
[10] ISO 18116, *Surface Chemical Analysis—Guide to Preparation and Mounting of Specimens for Analysis* (ISO, Geneva, 2004).
[11] ISO Standard 18117, *Surface Chemical Analysis—Handling of Specimens Prior to Analysis* (ISO, Geneva, 2009).

Published under license by AVS.

[12]ISO 20579-4, *Guidelines to Sample Handling, Preparation and Mounting—Part 4: Reporting Information Related to the History, Preparation, Handling and Mounting of Nano-Objects Prior to Surface Analysis* (ISO, Geneva, 2018).
[13]K. Siegbahn, Rev. Mod. Phys. **54**, 709 (1982).
[14]J. F. Watts and J. Wolstenholme, *An Introduction to Surface Analysis by XPS and AES* (Wiley, Chichester, 2020), p. 146.
[15]Transport of Ions in matter, see: www.SRIM.org.
[16]J. F. Watts and J. Wolstenholme, *An Introduction to Surface Analysis by XPS and AES* (Wiley, Chichester, 2020), pp. 97–131.
[17]A. G. Shard, F. M. Green, P. J. Brewer, M. P. Seah, and I. S. Gilmore, J. Phys. Chem. B **112**, 2596 (2008).
[18]N. Sanada, A. Yamamoto, R. Oiwa, and Y. Ohashi, Surf. Interface Anal. **36**, 280 (2004).
[19]I. Yamada, J. Matsuo, N. Toyoda, and A. Kirkpatrick, Mater. Sci. Eng. **R34**, 231 (2001).
[20]T. Miyayama, N. Sanada, S. R. Bryan, J. S. Hammond, and M. Suzuki, Surf. Interface Anal. **42**, 1453 (2010).
[21]D. R. Baer, D. J. H. Cant, D. G. Castner, G. Ceccone, M. H. Englehard, A. S. Karakoti, and A. Muller, *Characterization of Nanoparticles: Measurement Processes for Nanoparticles*, edited by V.-D. Hodoroaba, W. E. S. Unger, and A. G. Shard (Elsevier, Amsterdam, 2020), p. 316.
[22]A. Shchukarev, J. F. Boily, and A. R. Felmy, J. Phys. Chem. C **111**, 18307 (2007).
[23]P. G. Rouxhet and M. J. Genet, Surf. Interface Anal. **43**, 1453 (2011).
[24]A. Shchukarev and M. Ramstedt, Surf. Interface Anal. **49**, 349 (2016).
[25]L. Zhong, D. Chen, and S. Zafeiratos, Catal. Sci. Technol. **9**, 3851 (2019).
[26]R. M. Palomino, R. Hamyln, Z. Liu, D. C. Grinter, I. Waluyo, J. A. Rodriquez, and S. D. Senanayake, J. Electron Spectrosc. Relat. Phenom. **221**, 28 (2017).
[27]A. F. Gerwing, F. E. Doern, and W. H. Hocking, Surf. Interface Anal. **14**, 559 (1989).
[28]A. Zalar, Thin Solid Films **124**, 223 (1985).
[29]A. Zalar and S. Hofmann, Appl. Surf. Sci. **68**, 361 (1993).
[30]C. D. Easton, C. Kinnear, S. L. McArthur, and T. R. Gengenbach, J. Vac. Sci. Technol. A **38**, 023207 (2020).
[31]D. R. Baer, J. Vac. Sci. Technol., A **38**, 031201 (2020).
[32]K. Hellgardt and D. Chadwick, Rev. Sci. Instrum. **67**, 4025 (1996).
[33]ISO TR 22335, *Surface Chemical Analysis—Depth Profiling–Measurement of Sputtering Rate: Mesh-replica Method Using a Stylus Profilometer* (ISO, Geneva, 2007).

Published under license by AVS.