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DATA TREATMENT IN ELECTRON AND ION SPECTROSCOPY

W. L. BAUN

Mechanics and Surface Interactions Branch
Nonmetallic Materials Division

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Interim Report for period July 1977–July 1979

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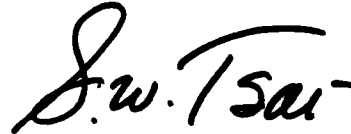
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

In order to fully utilize newly developed electron and ion surface spectroscopies, the data from these methods must be treated carefully to assure quality and clarity both during and after the experiment. Signal conditioning during the experiment puts the data into proper form for recording and may include steps for amplifying, digitizing, discriminating, modulating, gating, transforming and filtering. In Auger Electron Spectroscopy (AES) where modulation and synchronous detection methods are used, special tailored

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waveform modulation may be necessary to minimize elemental profile artifacts.
Dynamic background subtraction, digital filtering and Fourier transforms
improve the signal to noise ratio and facilitate further data processing.
If possible, an entire spectrum should be digitally stored at each plotted
point on the elemental depth profile. Such spectral storage allows retrieval
and examination of the spectrum for peak shape changes or appearance of peaks
which were not expected to occur. A point which must be considered is that in
bulk spectroscopies we may accumulate signal for whatever period is necessary
to obtain desired counting statistics while in surface spectroscopies using an
ion beam (such as ISS and SIMS), the eroding surface is ever changing and only
a finite time is available for counting at a given depth.

Following data acquisition and storage in digital form, several processing
steps may be used to enhance, simplify or otherwise make the data more useful
to the operator. These data presentation improvements include smoothing,
normalizing, differentiating, integrating and performing other mathematical
functions. An example of such a manipulation in AES is the integration of the
electron energy distribution curve $dN(E)/dE$ which recovers the energy distri-
bution $N(E)$. A second integration gives the original Auger electron current
which may in certain cases be more effectively used for quantitative purposes
than the usual peak to peak $dN(E)/dE$ values. Subtraction methods may be used
to emphasize the difference between a sample and standard or between a bulk
and a surface composition. Addition and subtraction of spectral features in
an interactive graphics system allows synthesis of any composition for
comparison with an unknown. Such methods also permit estimation of splotchy
surface coverage of thick or thin films.

Plotting subroutines produce high quality spectra and in addition simplify
great amounts of information and present it in the form of depth profiles,
histograms, or three dimensional plots which make the data more understandable
and clarify data trends.

FOREWORD

This technical report was prepared by W. L. Baun of the Mechanics and Surface Interactions Branch, Nonmetallic Materials Division, Air Force Materials Laboratory. The work was initiated under Project 2419, "Nonmetallic and Composite Materials" and WUD No. 44, "Improved Materials, Processes and Life Prediction Methodology of Adhesive Bonding" monitored by T. W. Haas.

This report covers work conducted inhouse during the period July 1977 through July 1979. It was published in Applied Surface Analysis, ASTM STP 699 T. L. Barr and L. E. Davis, Eds., American Society for Testing and Materials, 1980, pp. 125-137.

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W. L. Baun[1]

Data Treatment in Electron and Ion Spectroscopy

ABSTRACT: In order to utilize fully the newly developed electron and ion surface spectroscopies, the data from these methods must be treated carefully to assure quality and clarity both during and after the experiment. Signal conditioning during the experiment puts the data into proper form for recording and may include steps for amplifying, digitizing, discriminating, modulating, gating, transforming, and filtering.

After the acquisition and storage of data in digital form, several processing steps may be used to enhance, simplify, or otherwise make the data more useful to the operator. These data presentation improvements include smoothing, normalizing, differentiating, integrating, and performing other mathematical functions. An example of such a manipulation in Auger electron spectroscopy (AES) is the integration of the electron energy distribution curve, $dN(E)/dE$, which recovers the energy distribution, $N(E)$. A second integration gives the original Auger electron current which may in certain cases be more effectively used for quantitative purposes than the usual peak-to-peak $dN(E)/dE$ values. Subtraction methods may be used to emphasize the difference between a sample and the standard or between a bulk and a surface composition. The addition and subtraction of spectral features in an interactive graphics system allows synthesis of any composition for comparison with an unknown.

These techniques, combined with plotting subroutines, produce spectra of high quality and, in addition, simplify great amounts of information and present it in the form of depth profiles, histograms, or three-dimensional plots, which make the data more understandable.

KEY WORDS: surface analysis, data acquisition, digital methods, Auger electron spectroscopy (AES), ion scattering spectroscopy (ISS), secondary ion mass spectrometry (SIMS), X-ray photoelectron spectroscopy (XPS), ions, electrons

In order to utilize fully the newly developed electron and ion surface spectroscopies, the data from these methods must be treated carefully to assure quality and clarity both during and after the experiment. Signal conditioning during the experiment puts the data into proper form for recording and may include steps for amplifying, digitizing, discriminating, modulating, gating, transforming, and filtering. After the acquisition and storage of data in digital form, several proces-

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sing steps may be used to enhance, simplify, or otherwise make the data more useful to the operator. These data presentation improvements include smoothing, normalizing, differentiating, integrating, and performing other mathematical functions. The data treatment methods shown here apply equally well to all methods of surface characterization, including all of the methods typified in the familiar Propst diagram [1],[2] which shows the interaction on surfaces of electrons, neutrals, ions, photons, and various fields. Each of these surface probes may elicit a response, such as the emission of electrons, neutrals, ions, or photons, or a change in the field. That is, one may impinge electrons onto the surface, which may excite other electrons, as occurs in Auger electron spectroscopy (AES), or one may have photons exciting electrons on the surface, which is the basis of X-ray photoelectron spectroscopy (XPS), and each of these forms of spectroscopy may have subgroups, such as one in which a new particle may not necessarily be formed but the incoming particle may be changed. Therefore, there are dozens of possible forms of surface characterization as described by the Propst diagram.

Discussion

In these ion and electron spectrometries, as well as in other forms of surface characterization, the data treatment may be conducted during the experiment by signal conditioning, which includes amplifying, discriminating, modulating, gating, transforming, and filtering, and data treatment may be carried on after the experiment with various data operations such as smoothing, normalizing, differentiating, integrating, and performing other functions. In addition, some combinations of operations, both during the experiment and after the experiment, are frequently used for best data improvement. Table 1 shows combinations of analog and digital methods and the resulting versatility of each combination.

TABLE 1—*Combinations of data acquisitions and handling methods.*

Method and Versatility	1	2	3
Acquisition	analog	analog	digital
Handling	analog	digital	digital
Versatility	very limited	limited	unlimited

One near necessity in the handling of surface spectrochemical data is that the data be transformed into digital form and placed on some permanent record, such as magnetic tape or magnetic disks. Table 2 shows a comparison of analog

[2]The italic numbers in brackets refer to the list of references appended to this paper.

TABLE 2–*Comparison of analog and digital data acquisition and handling capabilities.*

Capability	Analog	Digital
Data evaluation	plot data	scaling, area, smoothing, math function, plot, etc.
Data capacity	limited, no storage	storage as required
Scan rate	several fixed values	unlimited
Calibration	manual	automatic
Diagnostics	manual	automatic (cables, lights, switches)

and digital methods in relation to several experimental parameters. There are numerous reasons for digitizing [2] in that all changes that take place as the surface is probed are permanently recorded. Also, there is no limit to the number of elements or peaks that can be recorded. It is not necessary to know in advance the changes that will occur or the new lines that may unexpectedly become visible. Alternate lines of the same or another series can be selected when overlapping or other conflicts occur. There are no off-scale peaks or off-scale profiles. Where elemental profiling is performed by ion milling techniques, one may go back and use chemical combination effects that can be monitored as a function of depth. These digital techniques allow for the automatic computer plotting of elemental profiles such as the curve shown in Fig. 1, the Auger profile of 2024 aluminum alloy. No surface preparation except for degreasing was carried out on this sample. It can be seen that the alloy as received has a very high magnesium concentration at the surface which drops off very rapidly with time. This magnesium-rich layer is very deleterious to adhesive bonding [3]. Samples bonded of this material, when exposed to long-time durability tests, show very poor performance. Other similar profiles also give a clue to the performance of various surface preparations such as the profile shown in Fig. 2, which is of a commercial acid etch performed, again, on 2024 aluminum alloy. In this particular case, the copper-rich layer, which lies in the region between the aluminum oxide on the surface and the bulk aluminum, varies considerably with the solution temperature and the time of etch [4]. This is an instance in which knowing the surface chemistry is not enough, but, rather, the elemental profile into the sample is required, since in some cases similar etches can cause the formation of weak boundary layers somewhat below the surface. These elemental profiles can be performed either by recording the entire spectrum, as suggested earlier, or by using a multiplex system, such as that available on several commercial surface characterization tools. The multiplex analog data presentation suffers the disadvantages outlined earlier. The data recording system used in this laboratory for Auger electron spectrometry is shown in Fig. 3, where a magnetic tape is used to record the entire spectrum. A modular approach may be used in a system such as this, where one uses only the particular modules needed. The most important factor is, again, to have sufficient memory to be able to record all of the spectrum, not just portions or peak values. An example of the utility of this method was found in a sample that was analyzed in this laboratory that was supposed to have
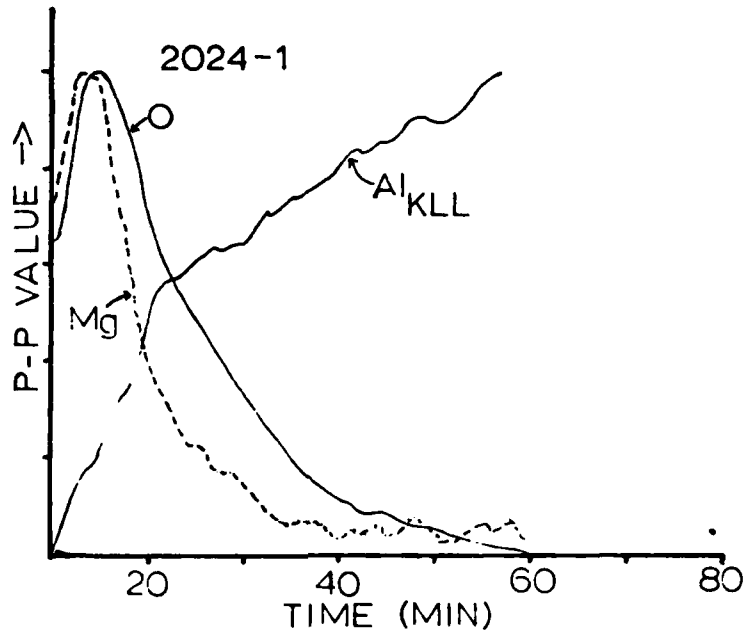
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FIG. 1–*Elemental depth profile using Auger electron spectrometry on 2024 aluminum alloy.*
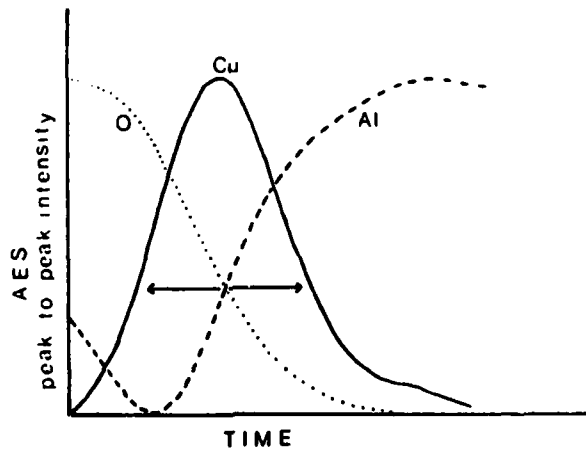

FIG. 2–*Elemental depth profile using Auger electron spectrometry on acid-etched 2024 aluminum alloy.*
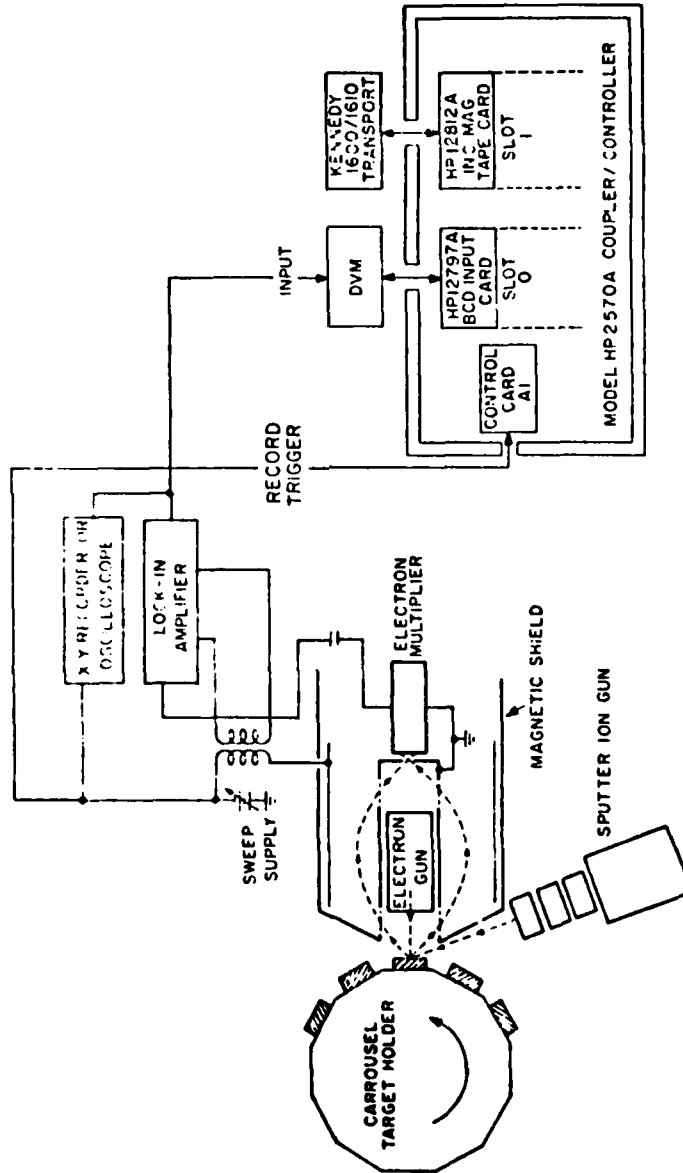
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FIG. 3 – Data recording system for Auger electron spectrometry.

a single antireflection coating on a quartz satellite window. As it turned out, this sample was eroded with an ion beam for over 600 min; it showed at least nine different coatings and seven different elements throughout the entire antireflection coating. With conventional multiplexing techniques, it would have been impossible not only to set up for each of these elements but also to obtain a single profile for each component in the complex coating. In addition, as the ion sputtering continues, there is very often a relatively large change in the characteristic lines in the spectrum. Very often these changes (which can be changes in shape or changes of line position) will cause problems in the elemental profile when it is plotted. Sometimes these shifts in the line position are so great that it takes the line position out of the region that the multiplex controller has been set to accept. Also, shape changes take place in profiling on aluminum oxide coating on aluminum. If one sets the multiplexer to accept a narrow region, then, by the time one has profiled or sputtered all the way through the film into the metal, the line has shifted considerably and the shape has changed. This produces a profiling artifact, which has been shown by Grant and coworkers [5]. This anomaly, seen in Fig. 4, in which the aluminum appears to drop in concentration as the interface between aluminum oxide and aluminum is reached, is entirely removed by special techniques, such as tailored modulation of the electron beam [6], synchronous detection, and integration of the data to recover the original Auger current.

In addition to using an ion beam to sputter the surface while the sample is actually investigated by an electron beam, as in AES, for instance, the interaction of the ion beam itself is often studied. This concept forms the basis for ion
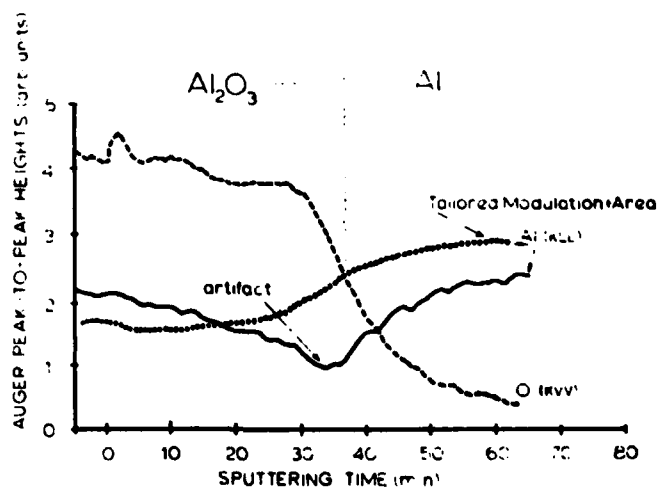

FIG. 4 - Elemental depth profile using Auger electron spectrometry, through aluminum oxide (Al₂O₃) on aluminum profiling artifact was removed by tailored modulation and integration of the signal.

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scattering spectrometry (ISS) [7]. The mechanism of this form of spectroscopy is described elsewhere in this volume and will not be repeated here. In addition to the direct collision of the impinging ion with the surface atom, which produces a single peak for each element on the surface, there is considerable new information being discovered on the ion scattering spectra, such as that found by Erikson and Smith [8]. These authors show that the ion yield curve, which had been considered to be smooth and featureless, actually contains a good deal of information that tells something about the chemical combination occurring at the surface. Christianson and coworkers [9], at the University of Minnesota, have shown that this oscillatory structure in the scattered ion yield curve for helium scattering from indium depends on the chemical environment of the indium. Very small changes that had been seen earlier in the spectra by Erikson and Smith [8] and by Rusch and Erikson [10] were found to be greatly enhanced and clarified by Fourier analysis of the oscillatory structure. In their method these authors digitized the data, then went through a preliminary processing step, and, finally, performed a fast Fourier transform, including smoothing and normalization of the data. Results of this Fourier transform for a group of indium compounds are shown in Fig. 5 and show striking differences due to the chemical combination of the indium.

In each of the surface spectroscopies simple filtering is frequently used to improve the signal to noise ratio. However, conventional filtering is effective only when the frequency spectrum of the signal and the frequency spectrum of the noise are not overlapping. A signal averaging computer (SAC), usually called a signal averager, is a very special kind of filter and is often used with surface spectroscopies. This method is sometimes referred to as a comb filter method, and it can be used effectively only if the desired signal, with its accompanying noise, can be generated a number of times. In most of the spectroscopies for surface analysis and, in particular, in ion scattering spectrometry, there are numerous reasons for using signal averaging. One very good reason is in order to record fast-changing phenomena, such as are found on very thin films which are eroded away rapidly, or to catch unstable species before they change on the surface. Another important use is the enhancement of peaks from elements present in very small concentration on the surface. This takes advantage of the improvement in peak-to-background ratio due to the repetitive scanning in the signal averaging method. Still another method has been semiquantitative analysis, using a subtraction method and an addition method [11]. Further, the signal averager is a convenient buffer storage for data which will be computer processed at a later date. The major advantage for signal averaging relies on the fact that for random noise the probable error decreases inversely to the square root of the number of signals measured. In theory then, noise can be reduced without limit. Of course, there are diminishing returns for continued efforts.

After the acquisition and storage of data in digital form, several processing steps, such as smoothing, normalizing, differentiating, integrating, and performing other mathematical functions may be used to enhance or simplify ion scat-

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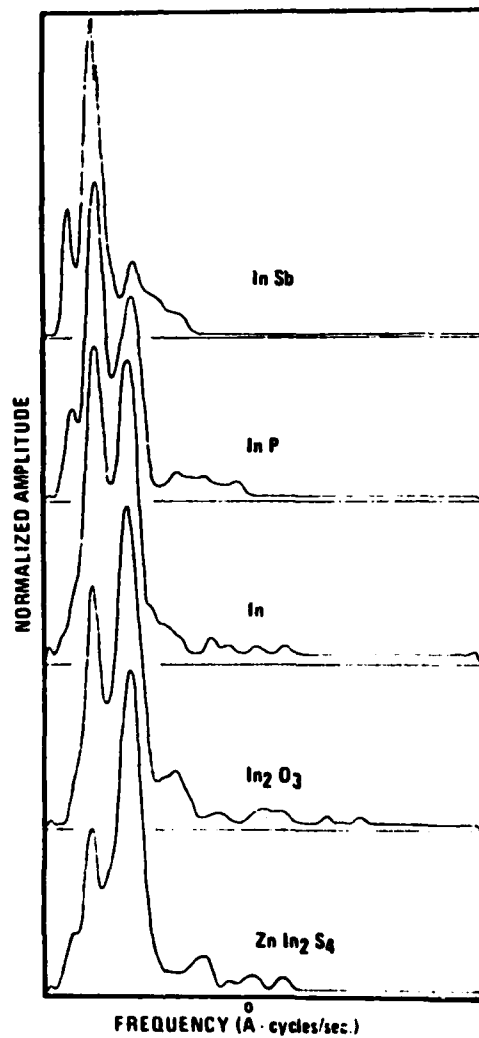
FIG. 5—*Ion yield curve structure from some indium compounds (from Christianson and coworkers [9].*

tering and many other surface spectroscopies. The routine normalization and smoothing of data results in uniform spectra that are more easily intercompared. In addition, certain segments of the spectra may be easily displayed in an expanded form, either in real time or plotted offline by the computer, as in the example shown in Fig. 6, which is an ion scattering spectrum of helium scattered from cobalt. A small amount of neon impurity, which scatters from cobalt, exists

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FIG. 6 *Ion scattering spectrum of helium scattering from cobalt, original and smoothed. (The asterisk shows cobalt (neon) resulting from residual neon scattering from cobalt.)*
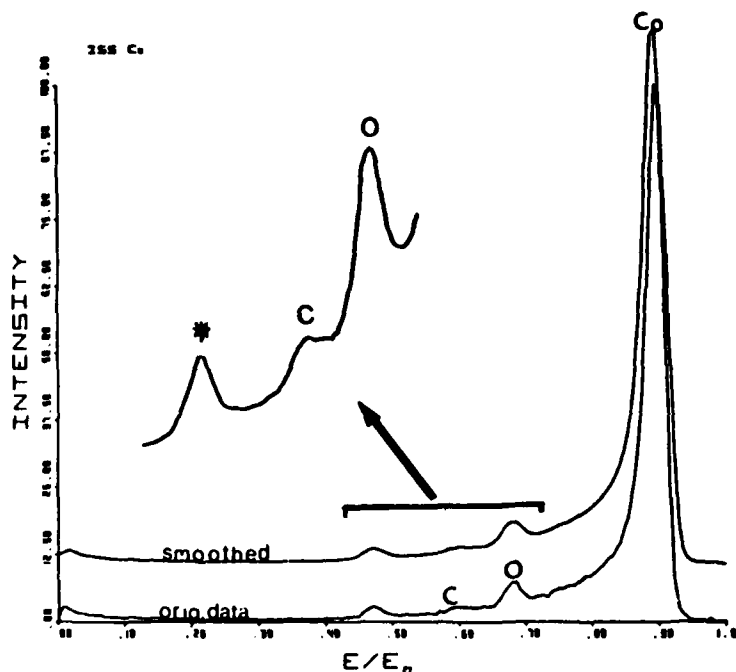
in the system. Such a large sloping background, seen in the expanded segment, can easily be removed, either during the experiment by choosing a narrow region and using dynamic background subtraction [11] or after the experiment by using subtraction techniques [12]. The recording of data in digital form is also of help in quantitative analysis. The obvious response of a researcher when told what chemicals are on the surface is to ask how much of each is present. Frequently, this question is not easy to answer for a variety of reasons. Perhaps, as Park says, it is a misapprehension that we should be able to define surface composition over a finite sampling depth. Some problems of little importance in bulk analysis are encountered in the quantitative analysis of surfaces [13] Sample matrix effects and physical phenomena such as roughness, selective diffusion, and grain boundary segregation play an important part in surface analysis. Factors originating with the measurement technique itself include selective sputtering, surface decomposition, changing ion and electron yields, and variable or unknown sampling depth. Still another problem is that one cannot count for an extended or unusually long period of time by using any technique with ion beam sputtering. Since the ion beam is constantly eroding away the surface, the sample composition is also constantly changing. In other forms of spectroscopy

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one may improve statistics, such as those described earlier for signal averaging, by counting for a longer period of time.

Digital data recording and reduction methods have been applied to the quantitative analysis of several binary alloy systems, using both ion scattering spectroscopy and Auger electron spectroscopy. Two methods have generally been used. The first and best method consists of using standard homogenous bulk alloy specimens which have been characterized by other techniques. The second method uses the bulk sensitivities of each element to allow calculation of the atomic fraction of each component, in a manner similar to the method of Flaim [14]. In this work Flaim introduces the concept of measuring elemental sensitivities in the equilibrium sputtered surface. The equilibrium sputtered surface is reached by sputtering the surface until no change is observed with further sputtering. This may not have the same composition as the bulk because of selective sputtering, but it is a reproducible surface standard. The method of elemental sensitivity may be applied quickly by using signal averaging. The unknown spectrum is recorded and stored in memory. Then, in another segment of memory, the pure materials that make up the alloy, such as copper and gold, are recorded in their pure form using, say, 100 individual signal averaging spectra, and then it is calculated just how many scans from each of the pure materials are necessary to synthesize the ratio observed in the original sample. In a similar manner, surface coverage where coverage is not complete may be calculated, as, for example, in a bonding experiment in which gold covered a titanium surface. The gold had been evaporated and then stripped off during a bonding test. Different surface treatments on the alloy left behind varying amounts of gold after the test, and the desired result was to be able to place a number on how much gold remained. Spectra representing incremental amounts of gold were recorded in the signal averager and subtracted from the original curve until no gold was visible in the spectrum, or, as described earlier, as an alternative method the incremental spectra from gold were added to a clean titanium alloy until the unknown spectrum was matched. By measuring how many counts of gold had to be added or subtracted, the coverage of gold could be calculated. Resolution can often also be enhanced by using subtraction techniques with digital data. Often lines from elements cannot be observed because they lie in the tails of other element lines. This background may be easily subtracted by using the techniques enumerated earlier. Such resolution enhancement has been shown earlier by this author to improve spectral resolution.

The methods described previously have been based primarily on analog-digital systems in which the data was recorded in digital form but the instrument control was analog. In all digital systems controlled by microprocessors or minicomputers, a great deal more versatility is possible. Such systems allow real-time display and smoothing of data and rapid expansion of spectral areas. The programming of these systems allows direct assignment of each feature from memory and comparison with standard spectra. It also allows for very versatile graphics, which produce high-quality computer-drawn spectra. The axis scales can be cal-

culated and drawn to any preselected scale. Any characters, subscripts, super-scripts, or special characters can be introduced into the graphics.

There is however, a bewildering array of hardware on the market. Each technical journal and trade magazine bombards the reader with the unparalleled advantages of his product. Sometimes it is difficult to tell just what a product will do in relation to one's particular problem. Even when the capabilities of a given product are known, it is hard to tell sometimes just how much computer or processing power is needed for a particular problem. Kessler [15] has developed a check list as a rough guide for assessing needs in a data acquisition system. This check list is shown in Table 3. Careful answers to the questions posed by Kessler will shorten the consideration time and quickly give an idea of just what is needed for a particular problem. The new emphasis in data acquisition is on the relatively low-priced microprocessor-based [16] data acquisition system. Such a system uses a large-scale integrated logic circuit on a chip (LSI) that controls the connection, sequencing, timing, measurement, input, output, and other functions of the system. This popular movement toward the use of microprocessors follows the trend in data processing of shifting from large centralized computers to arrays of smaller computers distributed where they are most needed. Guidelines are available from manufacturers' literature, several books which have been published in this general area [17,18], and journal articles.

In summary then, it can be said that physical and chemical properties of the surface play an important part in a large number of engineering and scientific applications. In order to use effectively the great amounts of data that are generated from surface analysis techniques, it is necessary to utilize automatic digital data acquisition and handling.

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TABLE 3–*Checklist for selecting a data acquisition system (after Kessler [15]).*

The following checklist is a rough guide for assessing your needs in a data acquisition system. Careful answers will shorten your consideration time and put you into the ballpark. Add the rating scores together and check the interpretive key below.

1. Recording function required
   - (a) Continuous ... 60
   - (b) Intermittent ... 40
   - (c) Not required ... 20
   - (d) Analog trend only ... 10

2. Scan rate required
   - (a) Real-time acquisition and processing ... 200
   - (b) 100 channels/s ... 100
   - (c) 25 channels/s ... 50
   - (d) Less than 5 channels/s ... 30
   - (e) Manual or fixed sampling rate ... 10

3. Data reduction required
   - (a) Instantaneous decision-making ... 100
   - (b) Multi-step (special function plus computer) ... 50
   - (c) Conversion to engineering units only ... 30
   - (d) Raw data printout only ... 10

4. Diversity of applications for the system
   - (a) Large dedicated real-time ... 100
   - (b) Multipurpose, changeable parameters ... 60
   - (c) Dedicated ... 40
   - (d) Multipurpose, fixed parameters ... 20

5. Personnel and computer available for operation of the system
   - (a) Virtually full-time access to data reduction computer and computer programmer ... 100
   - (b) Shared access to computer programmer with full-time computer available for data reduction ... 80
   - (c) Engineering-level personnel for system operation and data reduction and access to time-shared computer or equivalent ... 60
   - (d) Technician level system operator. Optional access to computer for tape analysis ... 40
   - (e) Hourly, unskilled level system operator. Direct data read-out from printer ... 20

Rating, Total Range	Type of Data Acquisition System
350 and up	computer-based
200 to 350	microprocessor-based
80 to 250	conventional
60 to 100	assembled from components
80 and below	multipoint recorder

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