

Using novel spectroscopy and spectrometry techniques for the quantitative analysis of photovoltaic thin films and materials

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This paper first appeared in the seventh print edition of *Photovoltaics International* journal.

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ABSTRACT

Chemical stoichiometry along with depth profiling and metallic contamination is of considerable interest for photovoltaic thin films. Conversion efficiency can be affected for example if primary components, e.g. Cd and Te, are not present at proper ratios. Moreover, amorphous silicon can vary substantially between sources and deposition technique, and qualitative comparison of trace metallic contaminants may not be sufficient to ensure final thin-film quality. This discussion presents data from atomic emission and mass spectrometry techniques that quantitatively and accurately describe both bulk and trace elemental compositions in photovoltaic materials, various thin-film matrices, and the final thin-film cell and module.

Introduction

Characterization of photovoltaic thin films is a wide-ranging subject that starts and ends with the all-important photovoltaic efficiency, and encompasses every aspect of the thin film and complete solar module. All of this characterization is needed simply to understand what properties enhance photovoltaic efficiency and how to improve it. As analytical chemists working traditionally for integrated device manufacturers in the semiconductor industry, it is normal to measure and quantify various materials and species concentrations. Examples include a chemical composition for a liquid chemistry used in process, or a contamination amount on the wafer surface that might affect properties of the final integrated circuit. Similar quantitative figures of merit have also

been identified and are needed within the photovoltaic industry where accurate measurement of compositional bulk elements, depth profiles of multiple elements, and determination of metallic species in thin films, different module layers, and the complete stack are required as technologies and processes evolve.

As briefly suggested by the term 'wide ranging,' chemical characterization for PV thin films is a topic where the authors give apologies to too many researchers and techniques that cannot be adequately referenced here. Impurities entrained in solar materials have long been recognized as a potential problem for effective photovoltaic conversion. Beginning with crystalline silicon (c-Si), neutron activation analysis (NAA) has been used for years [1], and glow discharge mass spectrometry (GDMS) more recently in an effort to

qualify silicon materials as suitable for PV applications [2]. Several studies have probed the required purity levels of crystalline silicon on ultimate photovoltaic performance [3].

“Impurities entrained in solar materials have long been recognized as a potential problem for effective photovoltaic conversion.”

With amorphous silicon (a-Si), the impurity levels are not as restrictive as with c-Si [4]; however, the presence of alkali and transition metals has been reported to affect solar cell performance

Constituent	NIST Standard			Glass-Powder	Soda-Lime Glass
	Certified value	Uncertainty	ICP-OES	ICP-OES	ICP-OES
	% (w/w)	% (w/w)	% (w/w)	% (w/w)	% (w/w)
SiO ₂	82.77	0.40	82.97	84.10	59.15
Fe ₂ O ₃	0.24	0.01	0.25	0.00	3.11
TiO ₂	0.11	0.02	0.12	0.00	0.09
Al ₂ O ₃	9.90	0.13	9.97	9.75	14.6
CaO	0.74	0.04	0.76	0.73	0.53
MgO	0.06	0.01	0.07	0.00	3.78
Na ₂ O	1.75	0.06	1.69	1.62	12.7
K ₂ O	3.94	0.09	3.90	3.90	3.94
BaO	0.12	0.01	0.13	0.00	0.02
B ₂ O ₃	N/A	N/A	0.00	0.00	2.12

Table 1. Compositional analysis of glass materials by ICP-OES indicates excellent agreement with NIST standards. Starting glass powder material and glass backing material for thin films can also be analyzed quantitatively via ICP-OES.

[5,6]. Ubiquitous alkali elements such as sodium and potassium are mobile ions and common transition elements including iron, nickel, and chromium can cause diffusion issues. All of these metals may be analyzed via depth profile studies and secondary ion mass spectrometry (SIMS) [6]. Beyond a-Si, polycrystalline thin films may also be affected by the presence of metallic ions. Sodium has long been understood as a positive influence species in copper-indium-gallium-(di)selenide (CIGS) films [7], while more recently iron diffusion behaviour in CIGS and zinc incorporation in CuInS₂ films have also been studied [8,9].

These are just a few examples and a few types of thin films where measurement of metallic species has been performed to understand ultimate solar cell performance. SIMS is one common technique for these types of analyses and provides real utility in its results. With SIMS however, different ionization conditions, ion sources, or mass spectrometers are needed to cover all elements of interest.

Measurement of trace metallic species, either through desired incorporation in a thin-film process or as an unwanted impurity, is just one measurement need for thin-film solar cells and modules. Compositional analyses in tandem with a depth profile of the thin film and/or conducting oxide material has been performed via a number of techniques and summarized recently [10]. In this case numerous techniques, including SIMS, Rutherford back-scattering spectroscopy (RBS), and energy dispersive x-ray spectroscopy (EDS), are used to analyze a CIGS film for Cu, In, Ga and Se distribution profiles. As shown, the results differ from technique to technique because of the different sensitivity factors associated with the measurement calculation. In order for engineers to be able to make direct comparisons of the results, it is highly desirable to perform these measurements against the same traceable calibration standards.

In this discussion, techniques are presented that can be used for quantitative measurement of metallic elemental species, overall elemental compositions, and depth profiles of various thin-film PV materials. Inductively coupled plasma optical emission spectroscopy (ICP-OES), RF glow discharge optical emission spectroscopy (RF GD-OES), and laser ablation inductively coupled plasma mass spectrometry (LA ICP-MS) are not traditional surface analysis techniques, and in fact have been used more prominently in non-high technology fields. However, by utilizing the inherent merits of each technique combined with simultaneous detection of elements across the periodic table, quantitative and comparable results are achieved. The result is a better understanding of some thin-film properties and an ability to solve specific thin-film issues.

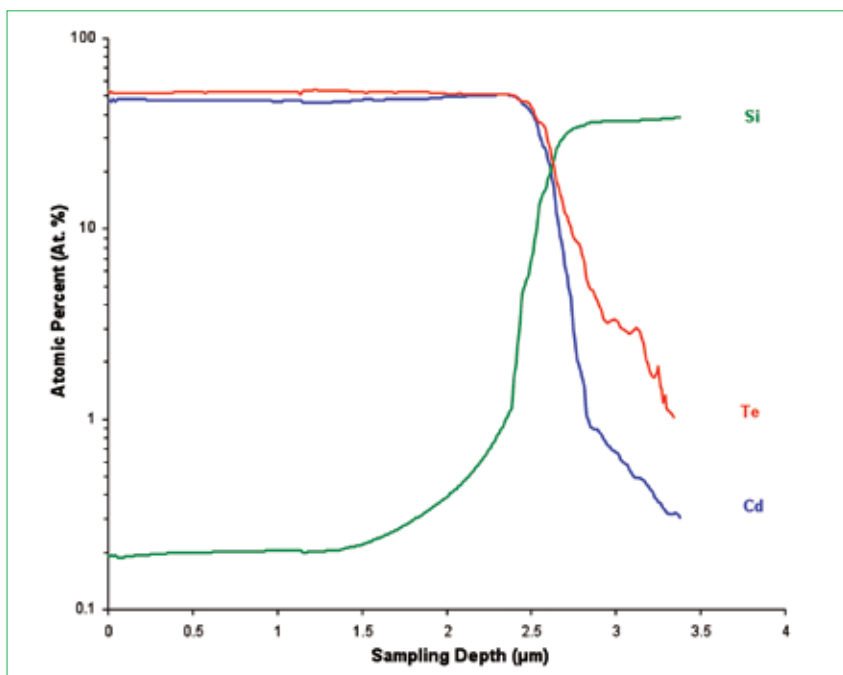


Figure 1. GD-OES depth profile for a CdTe film on silicate glass substrate.

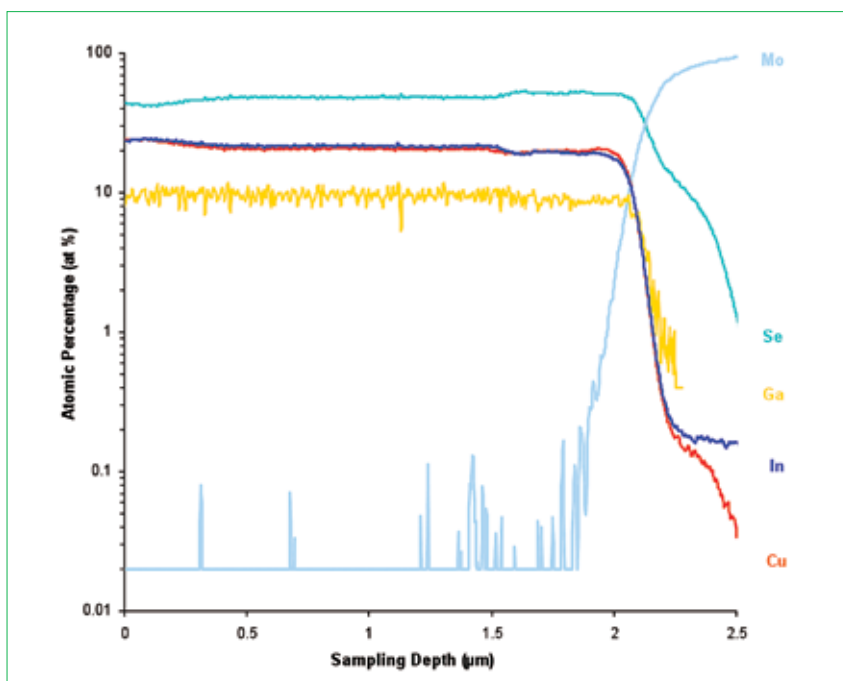


Figure 2. GD-OES depth profile obtained from a CIGS film on Mo-coated glass.

CIS Film	Cu at%	In at%	Se at%	In/Cu Mol Ratio	Se/Cu Mol Ratio
CIS-1	25.3	24.7	50.0	0.98	1.98
CIS-2	24.5	24.8	50.7	1.01	2.07
CIS-3	24.8	24.6	50.6	0.99	2.04
CIS-4	24.8	24.8	50.4	1.00	2.03
CIS-5	25.1	24.9	50.0	0.99	1.99
AV.	24.9	24.8	50.3	0.99	2.02
S.D.	0.29	0.12	0.34	0.01	0.04
RSD	1.2%	0.5%	0.7%	1.1%	1.8%

Table 2. ICP-OES measurements from five different film depositions indicate excellent reproducibility. Mole ratios for In/Cu and Se/Cu are another indicator of film quality.

Composition and stoichiometry verification

The composition of PV thin film and materials can be accurately verified using ICP-OES with both high accuracy and precision. Compared to atomic absorption spectroscopy (AAS), ICP has a large dynamic concentration range of up to 10^7 for many elements and permits simultaneous multielement determination without the need of a series dilution for high-concentration analyses. The sample preparation process consists of dissolving a thin film (or any applicable solid sample) into solution using mineral acids. This process generates a matrix solution congruent with commercially available calibration standards, such as those available from by the U.S. National Institute of Standards and Technology (NIST) and the national metrology institutes in Europe. As a result, the analytical results obtained by ICP-OES are generally independent of physical properties of the samples such as density, surface texture, film thickness, and crystal structure. This same ICP-OES technique is derived from applications used in the semiconductor industry where boron and phosphorus dopants are quantified in boron-phosphorus silicate glass films (BPSG), and these OES results are then used to calibrate XRF, FTIR, EDX, and SIMS [11].

It is certainly important to provide data and illustrate the quantitative nature of ICP-OES. Table 1 displays a comparison of ICP-OES experimental results with NIST-certified values and their uncertainties. With this type of agreement between experimental and theoretical data, actual samples can be analyzed with confidence. Results for a glass powder and soda-lime glass sample, a starting material, and final backing material for PV thin film, respectively, are also shown in the table. Different levels of impurities can easily be measured in this manner to ensure quality of the final glass substrate as well as prevent unwanted contamination in the final material.

Beyond the backing materials, PV thin films can be analyzed in exactly the same manner [12], and this analysis process has been utilized for CIGS, CdTe, CdS, CIS, and a host of III-V films. Table 2 shows that separate film depositions of a CuInSe₂ precursor film can be measured against one another with ICP-OES to show consistency between different processes. The results obtained are presented here in both atomic percentage and mol ratio from which the film stoichiometry can be calculated. It should be noted that Ga used in the CIGS to boost the film's light-absorbing bandgap can also be analyzed by ICP-OES with the same accuracy and precision.

A further application with ICP-OES has been used for determinations of major, minor, and trace elements in various electrochemical plating solutions. To lower the fabrication costs, thin-film PV manufacturers have investigated non-vacuum deposition approaches such as electrochemical deposition (ECD).

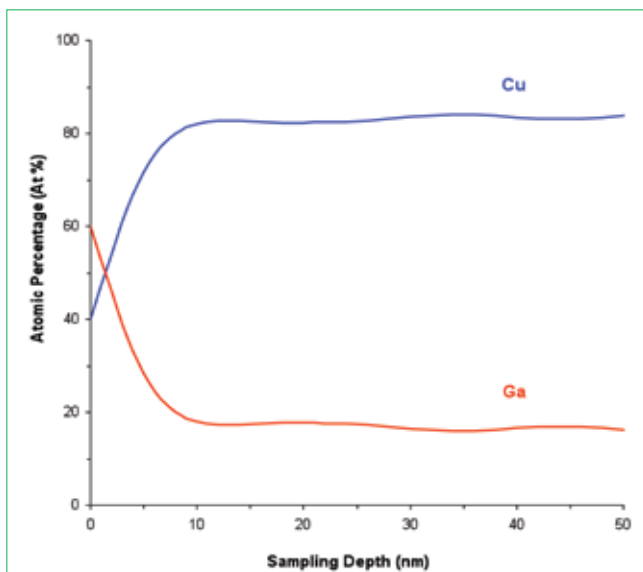


Figure 3. GD-OES depth profile obtained from a precursor CuGa film following chemical surface treatment.



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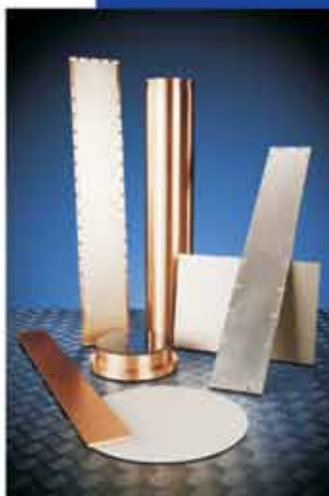
Sputtering Targets for Photovoltaics

Standard Materials Available



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Chromium
Copper
Indium
Molybdenum
Niobium
Nickel
Silicon
Tantalum
Tin
Titanium
Tungsten
Zinc
Zirconium



Alloys

Cd-Sn
Cu-In-Ga
Cu-In-Ga-Se
In-Sn
Ni-V
Si-Al
Ti-Al
Zn-Al
Zn-Sn
Zn-Sn-Sb

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Cadmium Telluride
Indium, Gallium & Copper Selenides
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Silicon dioxide
Titanium oxide TiOx
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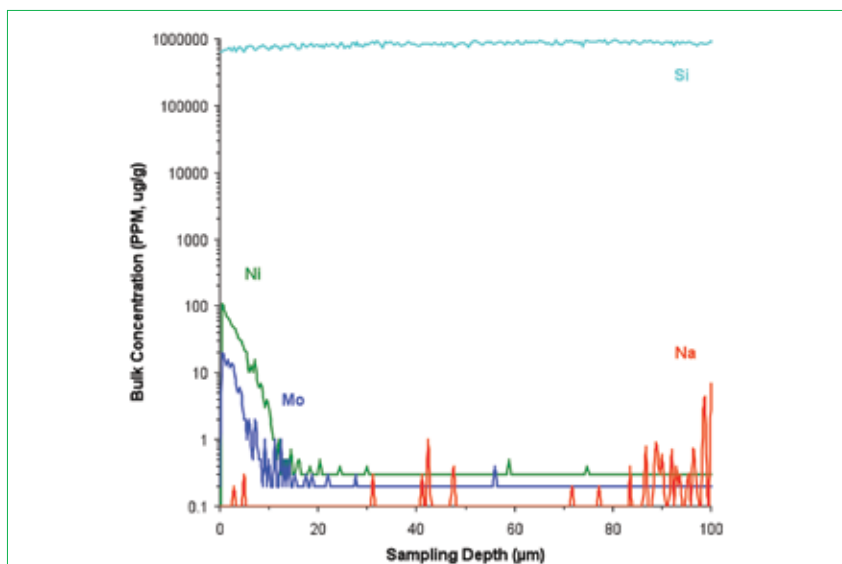


Figure 4. Deep depth profiling of an amorphous silicon thin film by LA ICP-MS.

Analyte	a-Si Film-1	a-Si Film-2	CuGa Film	CuGa Target
	ppm (μg/g)	ppm (μg/g)	ppm (μg/g)	ppm (μg/g)
Li	<0.05	0.06	<0.05	<0.05
Na	<0.01	12	2.4	2.2
Mg	<0.05	0.64	<0.05	<0.05
Al	<0.07	1.8	3.1	3.4
V	<0.01	0.05	<0.01	<0.01
Ti	<0.05	0.33	<0.05	<0.05
Cr	<0.05	4.2	<0.05	<0.05
Mn	< 0.5	1.3	< 0.5	< 0.5
Fe	<0.1	39	<0.1	<0.1
Co	<0.01	0.07	<0.01	<0.01
Ni	<0.05	2.7	<0.05	<0.05
Cu	<0.05	1.6	Major	Major
Zn	<0.1	0.9	1.7	1.6
Ga	<0.1	<0.1	Major	Major
Ge	<0.05	0.06	<0.05	<0.05
As	<0.05	0.08	<0.05	<0.05
Sr	<0.002	0.009	<0.002	<0.002
Y	<0.05	0.07	<0.05	<0.05
Zr	0.006	0.21	<0.005	<0.005
Mo	< 0.05	1.1	< 0.05	< 0.05
Ag	<0.01	<0.01	0.70	0.73
Cd	<0.05	0.08	0.34	0.38
In	<0.01	<0.01	0.04	0.04
Sn	<0.05	0.08	0.31	0.33
Sb	<0.005	0.01	<0.005	<0.005
Ba	<0.005	0.28	<0.005	<0.005
La	<0.01	0.05	<0.01	<0.01
Ce	<0.01	0.02	<0.01	<0.01
Hf	<0.01	0.02	<0.01	<0.01
Ta	<0.01	0.05	<0.01	<0.01
Tl	0.03	<0.01	<0.01	<0.01
Au	<0.02	0.07	<0.02	<0.02
Pb	<0.01	0.03	0.27	0.25
Bi	<0.005	<0.005	0.009	0.010
Th	0.12	<0.005	<0.005	<0.005

Table 3. LA ICP-MS results of a-Si solar thin films and CuGa film and corresponding sputtering target.

Many absorber layers (e.g., CdTe and CIGS) and window buffer layers (e.g., ZnO) can be electrochemically plated at reduced cost. The key to successful electrochemical plating is the quality of the plating bath chemistry; the concentration ratios of the film elements must be accurate and the unwanted impurities must be low. Because the ICP-OES results are consistent and reflect accurately any changes to the plating process, the use of this analytical technique for the quality control of plating bath solutions has been increasingly active for this type of thin-film PV manufacturing.

Elemental spatial distribution

An extension of compositional analysis is the need for depth profiling and elemental spatial distribution within the thin film. These types of data are important for both bulk elements, trace elements, and within both the thin film and solar cell. For traditional semiconductor thin films, SIMS is a workhorse for dopant profiling and its applications extend to other trace elements because of its high sensitivity. However, concerns exist with the ability of SIMS to profile solar thin films on a nonconductive glass substrate and the feasibility for quantitative analysis, especially of those major film elements because of its limited dynamic calibration range. In our laboratory, the elemental spatial distribution in PV thin films is typically studied using atomic spectroscopy and mass spectrometry, namely GD-OES and LA ICP-MS.

Glow discharge is an excitation source and owes its name to the luminous glow of the argon plasma. This plasma is generated by RF energy between an anode and a cathode sample surface and is followed by analyte atoms being knocked off the sample surface layer by layer. The sputtered atoms enter the GD plasma and collide with more energetic electrons or excited 'metastable' argon atoms to emit characteristic wavelengths of light [13]. Detection analyzers for a glow discharge source can vary as analyte ions can also theoretically be generated; however, the advantage of a polychromatic optical spectrometer is the available simultaneous multielement detection that in combination with the single-nanometer resolution in sputtering facilitates excellent depth-profiling capabilities.

Figs. 1 and 2 show GD-OES profiles obtained from a CdTe and a CIGS thin film, respectively. CdTe is deposited on a soda lime glass and CIGS on a molybdenum-coated glass substrate with a sampling depth in each case of more than 2.5 μm. No surface charging was present. The profiles shown in each figure were simultaneously obtained under a single sputtering condition. This is an advantage since real-time profiling ultimately eliminates the need

for overlaying different profiles obtained under different conditions. As a result, the association of those elements in the films and their corresponding concentrations can be directly investigated. Depth profiles obtained conveniently in this manner can be used to examine the film uniformity and possible interfacial diffusions, and to optimize CVD, PECVD, PVD, ECD, coevaporation, and other advanced deposition processes.

RF GD-OES can also be used to investigate the elemental concentrations on the top surface and in the near-surface region (e.g., < 20nm). The verification of surface composition of a thin-film postdeposition or after any surface treatment is extremely important for many reasons. One main reason is to ensure the interface integrity. For example, the band structure at the absorber-buffer interface or the p-n junction interface plays a crucial role in charge transport processes. Incorrect surface stoichiometry of one film alone can produce a poor band structure leading to low energy conversion efficiency and undesirable cell performance. In addition, the thin-film engineer needs evidence of minimal surface oxidation of the metal contact layer prior to deposition. Without this information, high contact resistance at the absorber-metal contact interface could lead to low photovoltaic efficiency.

Traditionally, Auger, SIMS, and XPS are used to study surface composition. However, the surface charging caused by the glass substrate, the surface equilibrium phenomena, and the thick information depth have prevented these techniques from revealing the true surface stoichiometry in such a challenging thin section. Fig. 3 provides a GD-OES depth profile for a CuGa precursor layer deposited using PVD (sputtering) after surface treatment. The sampling area used in the profiling was 4mm in diameter and the GD penetration depth was only 50nm. The CuGa target used for the deposition contains Cu (at 82%) and Ga (at 18%). As shown in the figure, the film and target compositions are fairly consistent in the 10-50nm region. However, the postdeposition surface treatment used in the process has dramatically altered the surface stoichiometry of the film by removing Cu and enriching Ga. As a result, the film composition on the top surface became undesirable, with Cu (at 40%) and Ga (at 60 %).

Previous GD-OES depth profiles have looked at bulk elements in the thin films. A higher sensitivity depth profile for trace elements is performed by LA ICP-MS. Laser ablation involves the conversion of a solid material into a plume of atomic vapour and microparticles by focusing a pulsed laser beam onto the sample surface. The plume is transported in an argon carrier gas to the inductively coupled

plasma for atomization and ionization. The ions produced by the ICP are then analyzed by either a quadrupole- or a magnetic sector-based high-resolution mass spectrometer [14,15].

Unlike analysis of secondary ions, LA ICP-MS does not use a primary ion beam, and spatially and temporally distinguishes its ionization from the sampling process. This separation enables independent optimization of the two fundamentally different processes, and generates much more efficient ionization and more quantitative measurements [15]. Because the impending laser beam does not carry any charge, PV thin films deposited on an insulating substrate or on conductive metal oxide films can be readily profiled without experiencing any 'surface charging' encountered with traditional electron and ion beam technologies. Finally, LA ICP-MS utilizes a fast scanning mass spectrometer and simultaneous detection is readily available for elements across the periodic table.

An example of a depth profile with LA ICP-MS is depicted in Fig. 4. In this case, a relatively large sampling area of 110 μ m and equally 'large' vertical sampling depth over 100 μ m was sampled. Trace element depth profiles of Mo and Ni near the surface and Na at ~100 μ m depth can be obtained from this very thick amorphous silicon film. LA ICP-MS has an ability to 'sputter' deep into the silicon bulk (e.g., > 50 μ m) to study the

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spatial distributions of trace impurities in the bulk of a film or a material. Such an analysis appears to be neither feasible nor practical for other traditional depth-profile techniques, such as Auger, XPS, and SIMS.

“With a spot-size analysis of $\sim 4\text{mm}$, the glow discharge sampling technique provides excellent sampling statistics for thin-film characterization.”

The OES and MS techniques have different advantages. A full rundown of each instrument is beyond the scope of this discussion; however, similar to ICP-OES, each technique allows simultaneous detection of elements across the periodic table. RF GD-OES provides improved depth resolution (single-nanometer resolution) along with analysis of metals and elements carbon, hydrogen, nitrogen, and oxygen. With a spot-size analysis of $\sim 4\text{mm}$, the glow discharge sampling technique provides excellent sampling statistics for thin-film characterization. Meanwhile, LA ICP-MS can analyze only metal and semimetals; however, the sensitivity of the ICP-MS surpasses that of OES (low parts per billion versus low parts per million), and the spot size with the laser ranges from 4 to $110\mu\text{m}$. When needed, a sampling area of $1\text{cm} \times 1\text{cm}$ is possible by rastering the laser beam across a thin-film surface. Both techniques can be applied to conductive and nonconductive surface materials, without surface equilibrium or charging issues at the sample or in the resulting data.

Trace elemental analysis

In order to maximize a solar cell's energy conversion efficiency and its viability, the quality of the films within the cell, such as absorber, buffer, antireflective coating, and front and back contact layers as well as the substrate, must be ensured. The verification of major film composition and its spatial distribution is described above; however, unintended impurities present in these films and coating materials must also be routinely monitored and controlled. Those impurities can adversely affect the cell's performance and can be detrimental to the lifetime of a PV module. All those films, coatings, and substrates can be analyzed for more than 80 elements by LA ICP-MS in a one-step elemental survey approach. The elements present in a material, including major ($>1\%$ w/w), minor ($0.01 - 1\%$ w/w), and trace elements ($<0.01\%$ w/w) can be positively identified and quantified. Because the power density of the laser spot on a

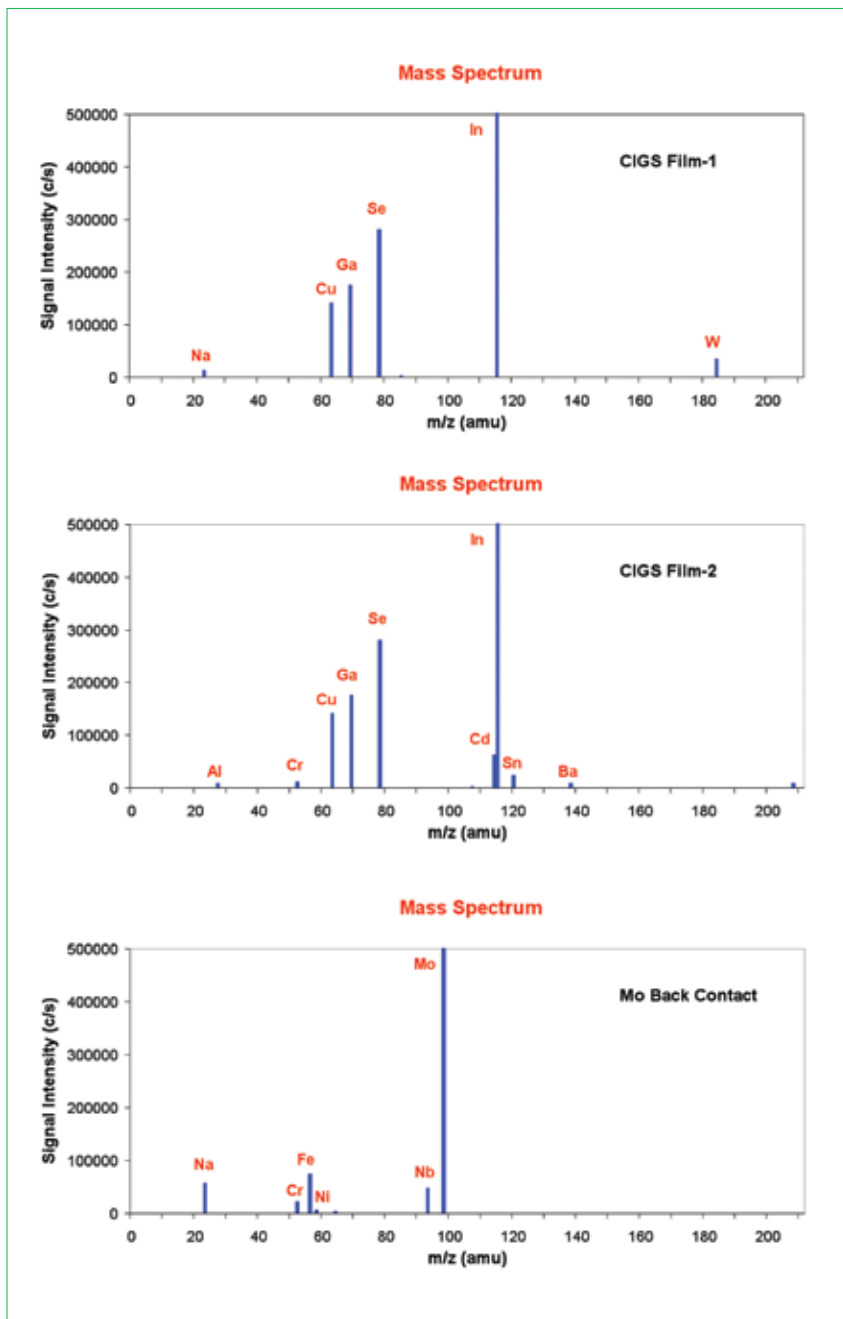


Figure 5. LA ICP-MS spectra obtained from two CIGS films and one Mo back contact.

material surface is so high ($\sim 10\text{GW}/\text{cm}^2$), any material, including inorganic and organic, conductive and non-conductive, refractory and fragile, can be analyzed by LA ICP-MS.

Fig. 5 shows three LA ICP-MS spectra obtained from three films deposited on a soda-lime glass substrate: two CIGS films grown under two different deposition processes and a commercially available molybdenum back contact film. It should be pointed out that these mass spectra were collected for more than 80 elements with either single or multiple isotopes. For simplicity in interpretation, only the major isotope for each element that was positively identified in the films is shown here. As can be seen in the figure, different deposition approaches can introduce different contaminants. These

contaminants could come from the deposition tools, target materials, or the handling process.

This LA ICP-MS elemental survey analysis has also been used to examine the components outside the cell but within a module for quality control purposes. These components include, but are not limited to: transparent top glass windows, encapsulation layers (usually thin sheets of ethyl vinyl acetate), and thin polymer rear layers (typically Tedlar) that prevent the ingress of water and gases, as well as the aluminium frame around the outer edge of the module.

In addition to the qualitative elemental survey, LA ICP-MS can also be used to perform quantitative analysis of various materials and films against NIST-traceable standards. This technique has been used

previously in the semiconductor industry for quantitative analysis of dopants in ultralow energy implants [16]. Table 3 shows the analytical data obtained from two amorphous silicon films, a CuGa precursor film and the sputtering target used to grow the CuGa film. A total of 68 elements were studied for these films and the concentrations of 35 important elements are provided in the Table. The other 33 elements were not detected at or above their corresponding detection limits (10 ppb for most of those elements). These consistent quantitative data, verified by NIST traceable standards, can help engineers select incoming raw materials, optimize film deposition processes, and track down the source of the contamination.

Conclusion

This article has presented surface analysis techniques and quantitative data generated for photovoltaic thin films, starting materials, and components within the solar module. Although not typically used for semiconductor or disk drive surface analyses, specific problem-solving for thin-film PV manufacturers has been achieved with the techniques presented here. Both LA ICP-MS and GD-OES are not new, as the laser technique has been used for close to 30 years with geology applications, and steel manufacturing and coating analysis has been the province of GD-OES. Despite such 'low-tech' beginnings, the techniques described here provide real data for PV thin films in R&D and manufacturing. No one technique is completely comprehensive for all analyses, but instead analytical tools must be used where a 'best fit' is realized and complementary in order to provide researchers and manufacturers the means to solve problems and improve PV efficiencies.

Other techniques will of course continue to serve for their specific advantages, for example SIMS for high-sensitivity depth profiling and Raman and FTIR for organic speciation, along with a host of other techniques and applications. Clearly, a phalanx of analytical, surface, and characterization techniques will be required as thin-film technologies continue to evolve.

Acknowledgments

The authors wish to thank **Wendy Rivello**, **Justin Laiduc**, and **Thao Nguyen** for their assistance in collecting the data presented here.

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