Analysis of Copper Indium Gallium Selenide (CIGS) Solar Cells with ARL QUANT'X EDXRF Spectrometer

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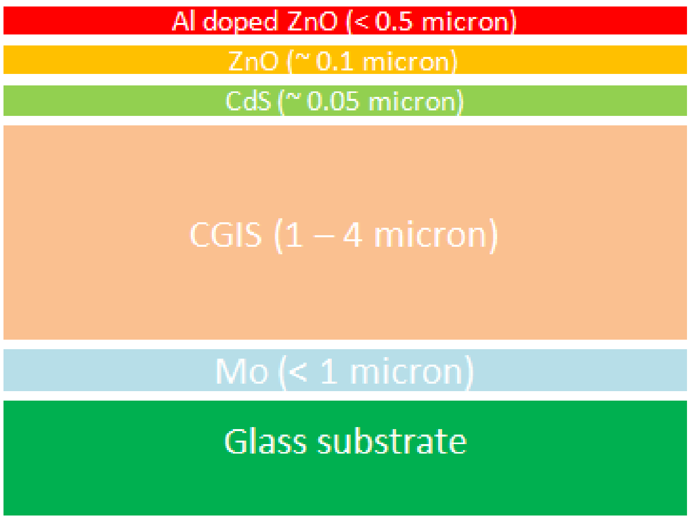
Introduction

Copper Indium Gallium Selenide (CIGS) is a direct bandgap semiconductor used in the manufacturing of solar cells. Because CIGS strongly absorbs sunlight, less material is required than for other semiconductor materials and this has led to the production of improved photovoltaics. A glass substrate coated with a Mo layer is used on which CIGS is deposited and sealed with layers of CdS, ZnO and Al doped ZnO. The Mo layer can be up to 1 micron thick while the thickness of the CIGS layer ranges from 1 to 4 micron. Energy-Dispersive X-ray Fluorescence (EDXRF) as a truly nondestructive technique is ideal for the characterization of CIGS solar cells, to determine both layer composition and thickness.

Figure 1: Cross sectional schematic of a typical CIGS solar cell.



Instrumentation

The Thermo Scientific™ ARL™ QUANT'X XRF Spectrometer is an EDXRF system which provides a fast and cost-effective analytical capability. It is fitted with an air-cooled Rh end-window tube with thin Be window (0.05 mm) and has a maximum power of 50 Watts. The ARL QUANT'X Spectrometer is equipped with an electrically cooled Silicon Drift Detector (SDD) with an area of 30 mm^2. The instrument features a total of nine primary beam filters ensuring that an optimal excitation condition is always found. An optional 10-position sample changer allows for unattended analysis.

Sample preparation and presentation

CIGS solar cells are analyzed as such without any sample preparation other than – if necessary – cleaning the surface of the cell. The spacious sample chamber can easily accommodate larger CIGS cells. Positioning the sample to analyze specific areas is facilitated by the availability of a CCD camera.

Excitation conditions

Table 1 shows the excitation conditions used for this application. As light elements are not present all measurements can be conducted in air. A measurement time of 30 s per condition will generate element characteristic peak intensities of several thousand counts. Figure 2 shows an example of a spectrum obtained from a CIGS solar module.

Table 1: Excitation conditions used to calibrate for and analyze CIGS solar cells.

Voltage (kV)	Tube Filter	Atmosphere	Live time (s)	Elements of Interest
30	Pd Thick	Air	30	Cu, Ga, Se
50	Cu Thin	Air	30	In, Mo

Calibration and validation

For the determination of both thickness and elemental composition of each layer we used the layer thickness and composition quantification method based on fundamental parameters (FP) included with the Thermo Scientific™ WinTrace™ software for ARL QUANT'X Spectrometer. This package only requires input about the number of layers and the elements present in each layer. Any information about thickness or concentration is not required.Calibration is done using bulk samples of Cu, Ga_2O_3, SeO_2, Mo and In as well as thin film standards for each element at a concentration of ~50 µg/cm² each.

Table 2 shows the analysis and repeatability results of a typical CIGS solar cell, and Table 3 shows a variant without any copper. Both cells were not covered with a transparent front contact explaining the absence of any Cd and Zn (see also Figure 2). As the glass substrate contains no elements having any enhancing effects on the CIGS elements and Mo, its effect can be discarded and the stack to model essentially consists of a Mo layer and a CIGS layer. The repeatability of the instrument/method was tested by analyzing both samples 10 times in a row. At a relative error of 0.5% or less, the repeatability of the layer thickness determination is excellent. Relative errors on the concentrations are below 1% for all elements except for Ga (1.5%). Increasing the measurement time of the 30 kV excitation condition to 2 minutes would also decrease the RSD value for Ga well below 1%.

Figure 2: Spectrum of a CIGS solar cell acquired with an excitation voltage of 50 kV and a thin Cu filter. The measurement time is 30 s, sufficient to generate intense element characteristic peaks.

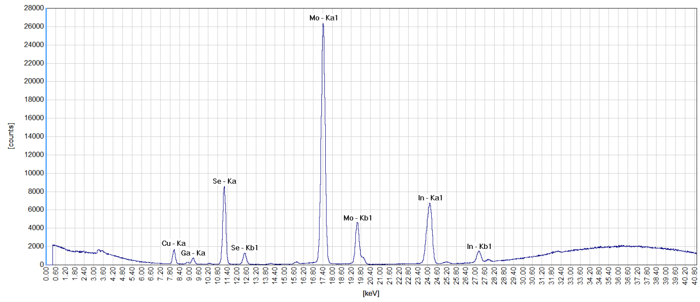


Table 2: Analysis and repeatability results of a typical CIGS solar cell.

Sample Run #	CIGS layer					Mo layer	
	CIGS (µm)	Cu (%)	Ga (%)	Se (%)	In (%)	Mo (µm)	Mo (%)
CIGS A01	1.292	16.26	5.90	49.91	27.92	0.925	100
CIGS A02	1.303	16.42	5.74	49.92	27.92	0.922	100
CIGS A03	1.289	16.17	5.82	49.70	28.31	0.922	100
CIGS A04	1.294	16.22	5.91	49.60	28.28	0.922	100
CIGS A05	1.293	16.26	5.79	50.10	27.86	0.934	100
CIGS A06	1.295	16.38	5.90	49.76	27.96	0.924	100
CIGS A07	1.298	16.31	5.91	49.66	28.12	0.927	100
CIGS A08	1.301	16.04	5.71	49.72	28.54	0.926	100
CIGS A09	1.290	16.24	5.84	49.71	28.22	0.934	100
CIGS A10	1.294	16.29	6.02	49.98	27.71	0.931	100
Average	1.295	16.26	5.85	49.81	28.08	0.927	100
1-Sigma	0.005	0.11	0.09	0.16	0.25	0.005	0

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Table 3: Analysis and repeatability results of CIGS type solar cell containing no copper.

Sample Run #	CIGS layer					Mo layer	
	CIGS (µm)	Cu (%)	Ga (%)	Se (%)	In (%)	Mo (µm)	Mo (%)
CIGS B01	1.103	0.06	7.19	53.13	39.62	0.931	100
CIGS B02	1.121	0.04	7.31	52.82	39.84	0.940	100
CIGS B03	1.119	0.00	7.40	52.61	39.99	0.941	100
CIGS B04	1.113	0.06	7.11	52.71	40.12	0.934	100
CIGS B05	1.117	0.14	7.36	52.80	39.71	0.933	100
CIGS B06	1.114	0.03	7.30	52.97	39.70	0.939	100
CIGS B07	1.118	0.11	7.27	52.54	40.09	0.935	100
CIGS B08	1.117	0.03	7.36	52.48	40.13	0.940	100
CIGS B09	1.123	0.09	7.35	52.48	40.09	0.943	100
CIGS B10	1.119	0.10	7.27	52.50	40.14	0.932	100
Average	1.116	0.06	7.29	52.71	39.94	0.937	100
1-Sigma	0.006	0.04	0.09	0.22	0.20	0.004	0

Conclusion

This application note shows the excellent performance of the ARL QUANT'X EDXRF Spectrometer and its SDD for the non-destructive analysis of CIGS solar cells. Total measurement times of 1 minute live time allow for excellent repeatability values, both for the layer composition and thickness. The FP based thin film software allows measurement of thickness, mass and composition of up to 6 layers containing any number of elements.

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