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Design and Implementation of an Experimental Setup for Automated Photothermal Spectroscopy

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Abstract: Recent developments in solar energy research have led to new technologies in solar cells. Materials based on chalcopyrite ternary semiconductors have gained great interest because of their excellent optical properties. It has been shown that the compound's defect structure affects strongly the optoelectronic properties of these alloys. In order to obtain more efficient devices these defects need to be detected and identified. The best way to characterize those novel materials is to use new techniques based on the exploitation of the photothermal effect. The main objective of this work is to study and implement an automated photothermal spectrometer. The first measurement technique that can be done with this apparatus is the photoacoustic spectroscopy. Wavelength scanning and data acquisition and logging are done automatically by a program written in LabVIEW. The second measurement technique is a transition from the former one. It is done by modifying only a few devices. Thus, one can make the photothermal beam deflection spectroscopy simply by adding a laser source and the position sensor detector that goes with it. The last measurement technique that can be done with the implemented setup is photothermal imaging analysis. We just add a motorized X-Y stage and a suitable focusing optics. The assembled apparatus conceived in this work constitutes an essential tool for analyzing and characterizing ternary and quaternary absorbing materials for photovoltaic and microelectronic applications. This setup can also be used by a large number of scientists in various disciplines such as biology, medicine, physics and optics, since the analysis is nondestructive and without contact. It provides researchers with information on non-radiative de-excitation processes regardless of the form of the samples (solid, liquid, gel or gas). Finally, the test and calibration procedures necessary to have a functional and accurate system are reviewed.

Keywords: Photothermal, Spectroscopy, Photoacoustic, Chalcopyrite, Characterization

Introduction

Recent developments in solar energy research have led to new technologies in solar cells. The developments based on ternary semiconductors have aroused great interest because of their excellent optical properties. The use of solar cells heterostructures obtained by deposition of thin layers by low cost process is considered as a priority research track to optimize the cost of manufacture / yield. In addition, it has been shown that the properties of the different layers and the different interfaces are mainly responsible for limiting the photovoltaic conversion. Several phenomena remain unexplained, in particular the phenomena of recombination in such structures. The best way to characterize them is to use a new technique based on the exploitation of the absorption spectrum obtained by photoacoustic spectroscopy (PAS). [Rosencwaig]

The technique of photoacoustic spectroscopy (PAS) has shown a very important development in several directions in recent years. It is used by a large number of scientists in various disciplines such as biology, medicine, physics, electronics and optics [Sell]. PAS is unique in that it provides researchers with information on non-radiative de-excitation processes with the potential to give deep profiles of samples regardless of their form (solid, liquid, gel and gas) [Zegadi1]. The analysis is nondestructive and without contacts. The spectrum

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obtained is based on the heating of the material considered as the origin of the greatest loss in the conversion process, therefore, it will analyze the channel responsible for these losses.

In this work we will go through the steps of the development of the photothermal spectrometer intended for material characterization especially for photovoltaic and microelectronic applications. The different variants of this system will also be exposed. Finally, the test and calibration procedures necessary to have a functional and accurate system are reviewed.

We want to mount a spectrometer that allows us to do the three spectroscopic techniques related to the photothermal effect which are: photoacoustic spectroscopy, photodeflection spectroscopy and photothermal imaging.

Photoacoustic Spectrometer

The photoacoustic spectrometer is based on the irradiation of a sample with a modulated monochromatic light source (Figure 1). A lens transforms the divergent beam of the lamp into a beam of small diameter. The latter is modulated by a mechanical optical chopper. The monochromator is used to disperse this luminous radiation, thus allowing to select the excitation wavelength. After passing through a filter (elimination of unwanted wavelengths), an elliptical mirror focuses the light beam thus obtained on the sample to be studied which is placed in the photoacoustic cell.

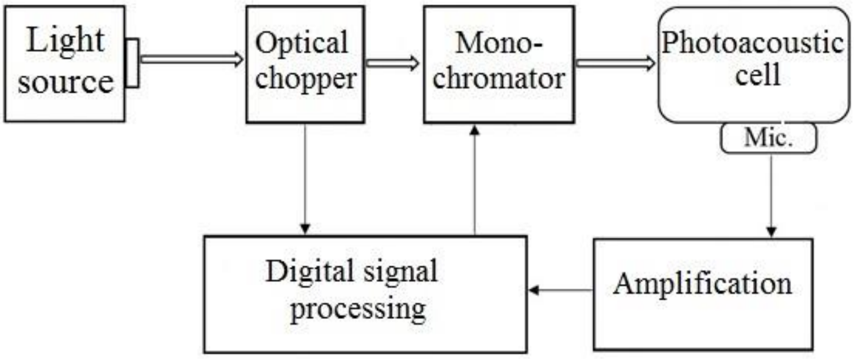


Figure 1. Synoptic diagram of the system [Zegadi2]

As a consequence of the absorption, the interaction of the surface layer of the sample is manifested by a thermal release which produces pressure fluctuations, which would result in a photoacoustic signal. The latter can be detected either by an acoustic or piezoelectric sensor. This low-amplitude signal is then amplified using a high-sensitivity AC amplifier and then extracted from noise by a lock-in amplifier and analyzed by a computer using the various signal processing methods.

Each instrument or element of the assembly must be appropriately chosen according to its possibilities and characteristics so as to construct a homogeneous assembly which fulfills the function for which it is designed and thus to obtain reliable and reproducible data. In the following, we detail the choice and the usefulness of each instrument of the assembly.

Excitation source

The sources of electromagnetic radiation that can be used are of two types: sources with a wide spectral range and narrow band ones (laser sources). For an analysis that requires wavelength variation, it is preferred to use the first type of source. For other analysis one opts for a laser source (continuous or pulsed).

In our assembly we use a radiation source with a wide spectral range: a short arc Xenon halogen lamp with a power of 300 W. A lens transforms the divergent beam of the lamp into a spot that fits between the slots of the chopper wheel.

Modulator (Optical Chopper)

The light beam is modulated ON-OFF using a variable frequency mechanical optical chopper. The model used is a MC2000 from Thorlabs (Figure 2). The wheel that comes with the chopper allows frequencies ranging from 20Hz to 1000Hz. The reference output signal is set to the actual frequency of the modulation wheel. This signal is injected into the lock-in amplifier to facilitate the extraction of the useful signal embedded in noise.

Figure 2. Head and wheel of the mechanical optical chopper

Monochromator

To select the excitation wavelength, the input light beam is dispersed using a high precision monochromator. The chosen model is the TRIAX 180 from HORIBA Scientific. The latter has an aperture of f/3.9 with a focal length of 19 cm and a spectral dispersion of 3.53 nm/mm. The monochromator has motorized adjustable slots located at the input and output ports (0-7 mm in 6.25 μm increments). It also has a triple turret dispersion network. The network of 1200gr/mm allows a spectral range of 0-1400nm.

Filter and focalization

The modulated monochromatic beam is then filtered using a filter that only passes light of wavelengths close to the region of interest. The beam is then directed using an elliptical mirror f/0.7. This serves to focus the light beam so that the incident ray only illuminates the desired part of the sample.

Photoacoustic cell

The photoacoustic cell is the chamber containing the sample to be studied as well as the microphone or transducer. It represents the heart of the spectrometer and directly influences the measurements made on different types of samples. All theoretical studies predict that the amplitude of the PA signal reaching the microphone depends on the size of the cell. In general, the amplitude of the PA signal increases when the dimensions of the cell are reduced. Different forms of PA cells have been reported in the literature [Zegadi2]. These cells can be classified according to the sample to be studied: cells designed for gaseous samples and others for solid samples. For the first case, the most used detector is the microphone. Regarding the second case one can use either a microphone or a piezoelectric sensor or both simultaneously.

The method of indirect detection is based on the thermal coupling from the sample to the gas column, the detector used is a microphone. This method was chosen for our assembly. The direct detection method uses a piezoelectric transducer as a sensor. The latter offers a greater range of modulation frequencies. Figure 3 shows a diagram of a photoacoustic cell. The window material is usually transparent to the studied wavelengths.

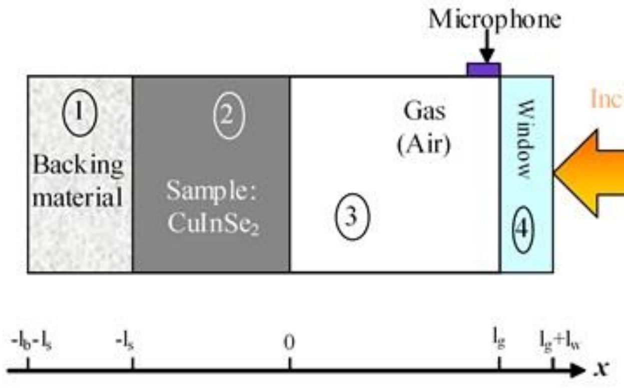


Figure 3. PA cell diagram

The study and design of photoacoustic cells is necessary for a good measurement of the photoacoustic signal. The cells may be parallelepipedal or cylindrical. The cell material may be stainless steel or plexiglass. Two examples of PA cells are shown in figure 4.

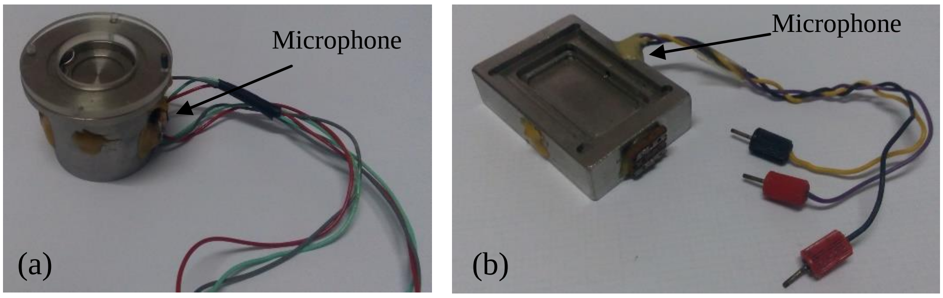


Figure 4. Photoacoustic cell a) cylindrical b) parallelepipedic

We chose a high sensitivity microphone to detect the generated PA signal. It is model BT1753, manufactured by Knowles Electronics. It is attached to the outside of the cell using a resin to prevent any kind of gas leakage or gas exchange between the inner part of the cell and the outside. It is connected through a channel leading to the sample compartment, to avoid any contact with light that might affect the signal.

Preamplifier and Lock-in

The output signal from the microphone (PA signal) is of very low amplitude. A high sensitivity AC amplifier amplifies this signal. The SR560 model from Stanford Research Systems was chosen.

The preamplified photoacoustic signal can be embedded in noise. A synchronous detection amplifier (lock-in) is used to separate the wanted signal from the noise. It is the model LI5640 from NF-corporation that was chosen. It is a digital multifunction lock-in amplifier in which high stability is achieved by a DSP processor. The instrument has a base capacity for frequencies ranging from 1mHz to 100KHz and a voltage sensitivity ranging from 2nV to 1V at full scale.

Signal processing

A microcomputer (PC) is used for storage and processing of the measured data. The same PC Controls the entire measurement system (optical chopper, monochromator, lock-in). Data acquisition is done through the IEEE 488 interface (GPIB). The exploitation of the photoacoustic signal, i.e analysis of the in-phase and in-quadrature signals as a function of the wavelength at a fixed modulation frequency, makes it possible to deduce certain optical properties from the sample under test. Figure 5 shows the mounted photoacoustic spectrometer. The optical chopper is connected to the PC through USB interface. The monochromator uses IEEE 488 interface.

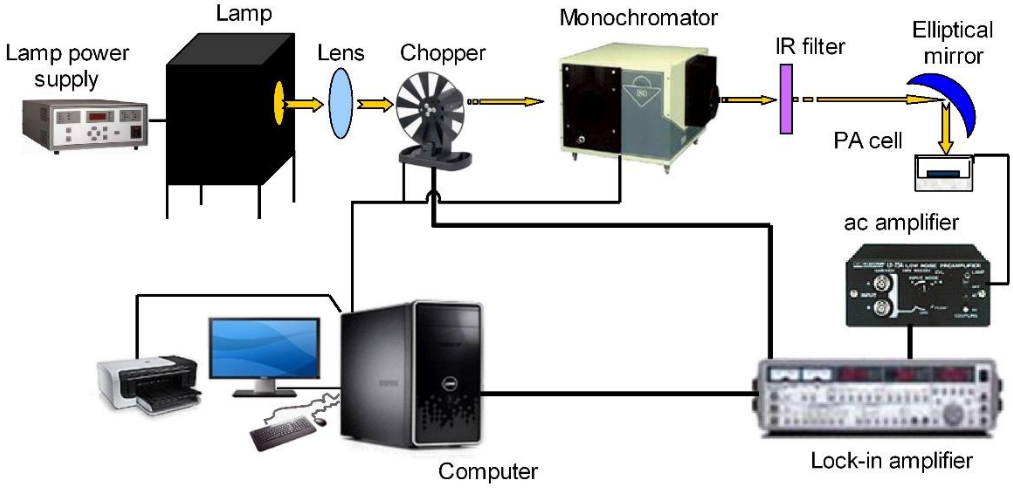
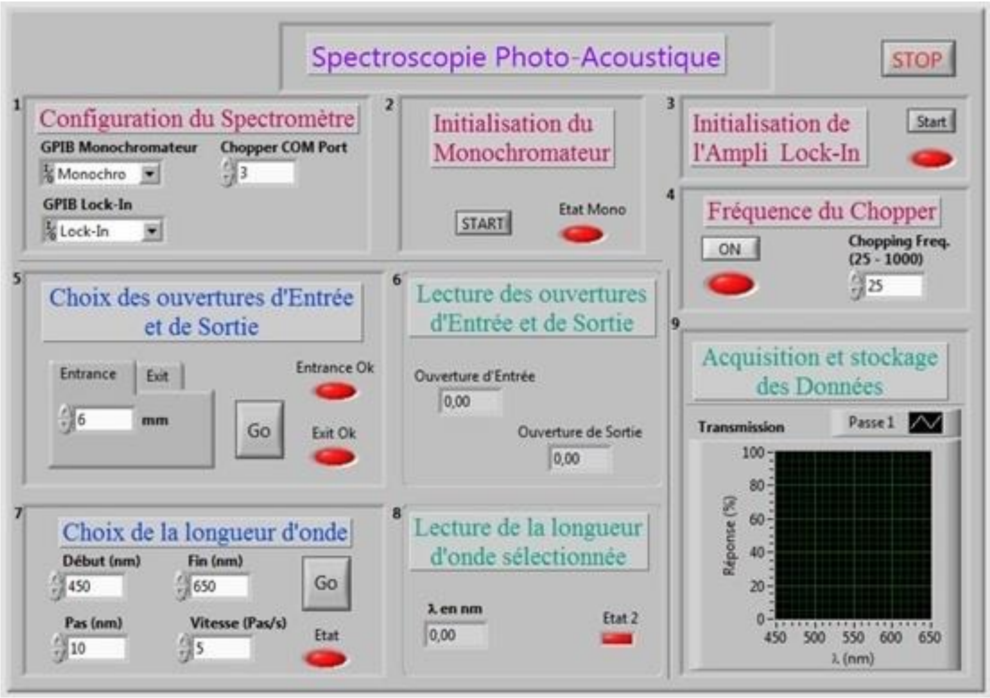


Figure 5. The photoacoustic spectrometer

Control software

The various instruments shown in Figure 5 are controlled by the famous LabVIEW language from National Instruments. LabVIEW is typically used for data acquisition, instrument control, and industrial automation. It can support interfaces such as RS232, GPIB, USB, etc. A control program has been written using version 17 of LabVIEW. Figure 6 gives an overview of the front face of our spectrometer while figure 7 gives the block diagram which is the graphical code itself.


Spectroscopie Photo-Acoustique

1 Configuration du Spectromètre
GPIB Monochromateur: Monochro
Chopper COM Port: 3
GPIB Lock-In: Lock-In

2 Initialisation du Monochromateur

3 Initialisation de l'Ampli Lock-In

4 Fréquence du Chopper
Chopping Freq. (25 - 1000): 25

5 Choix des ouvertures d'Entrée et de Sortie
Entrance: 6 mm

6 Lecture des ouvertures d'Entrée et de Sortie
Ouverture d'Entrée: 0,00
Ouverture de Sortie: 0,00

7 Choix de la longueur d'onde
Début (nm): 450
Fin (nm): 650
Pas (nm): 10
Vitesse (Pas/s): 5

8 Lecture de la longueur d'onde sélectionnée
λ en nm: 0,00

9 Acquisition et stockage des Données

Figure 6. Front panel of the Virtual Instrument Spectrometer PAS

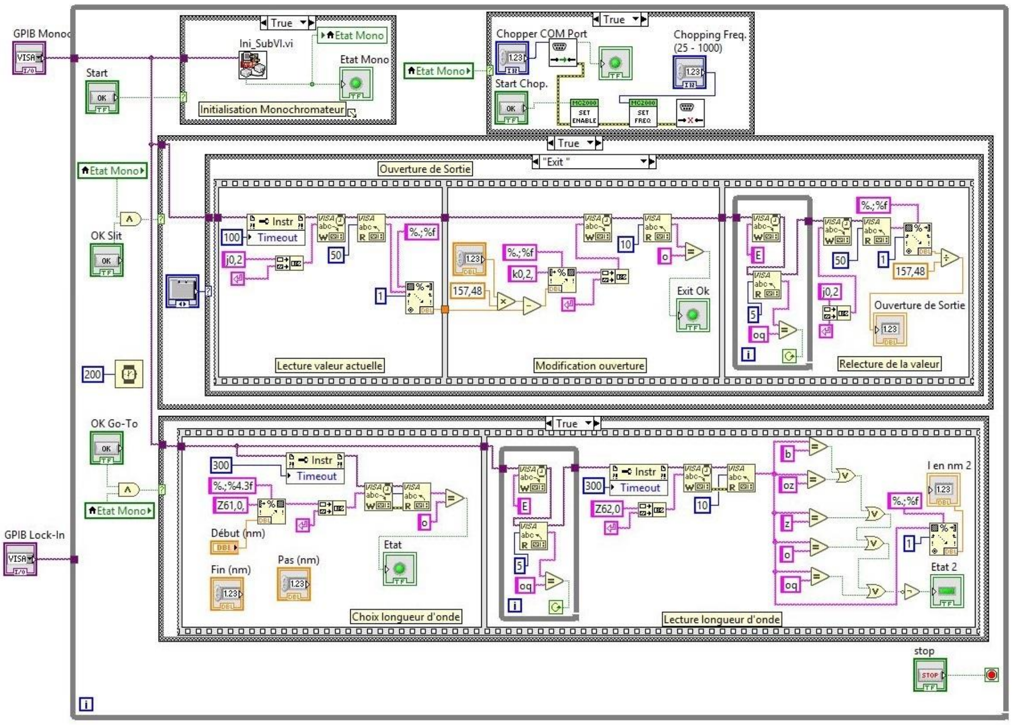


Figure 7. LabVIEW block diagram (graphical program) of the PA Spectrometer

Photodeflection Spectrometer

The principle of photodeflection spectroscopy (PDS) is as follows: a sample is irradiated by a modulated (periodic or pulsed) monochromatic light beam. The energy released as a result of the absorption of light causes a variation of certain physical parameters of the sample itself or the surrounding environment. A probe beam traversing this medium will be deflected and the amplitude of the deviation and its phase are a function of the thermal and optical properties of the sample under test. Figure 8 shows a block diagram of a photodeflection spectrometer using the principle just mentioned.

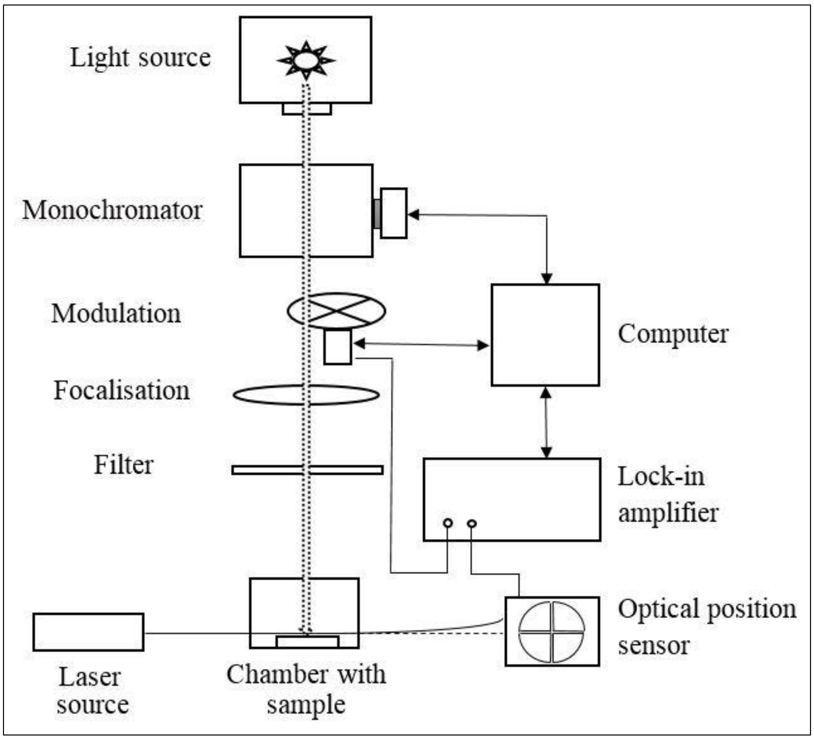


Figure 8. Block diagram of a PDS system " mirage " [Bocara]

Figure 9 illustrates a diagram of the cell containing the test sample and the surrounding fluid.

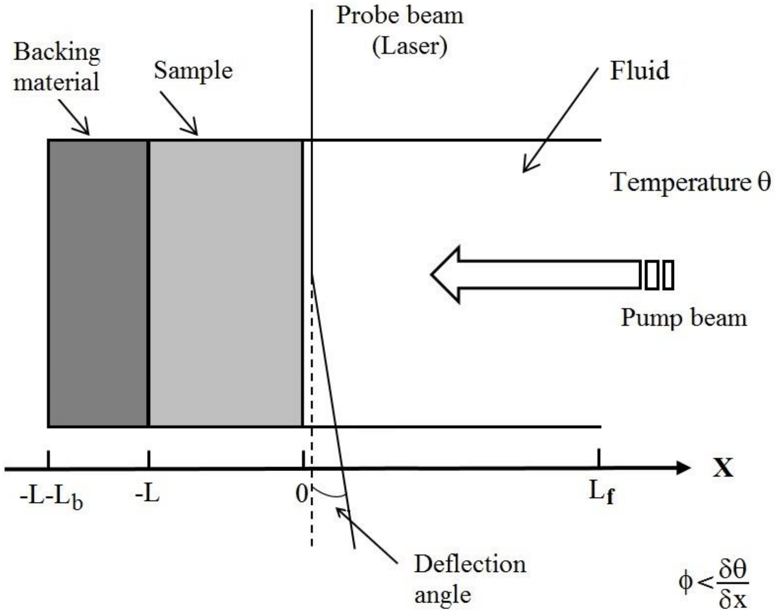


Figure 9. The geometry of a one-dimensional cell

The same setup used in photoacoustic spectroscopy can be used here with some small modifications. The excitation source can remain the same as it can be replaced by a laser source (continuous or pulsed). The sample cell must be transparent to let the laser probe beam passes through. The microphone is replaced by a position sensitive device which is often a 4-segment photodiode detector. Figure 10 depicts the assembled photodeflection spectrometer.

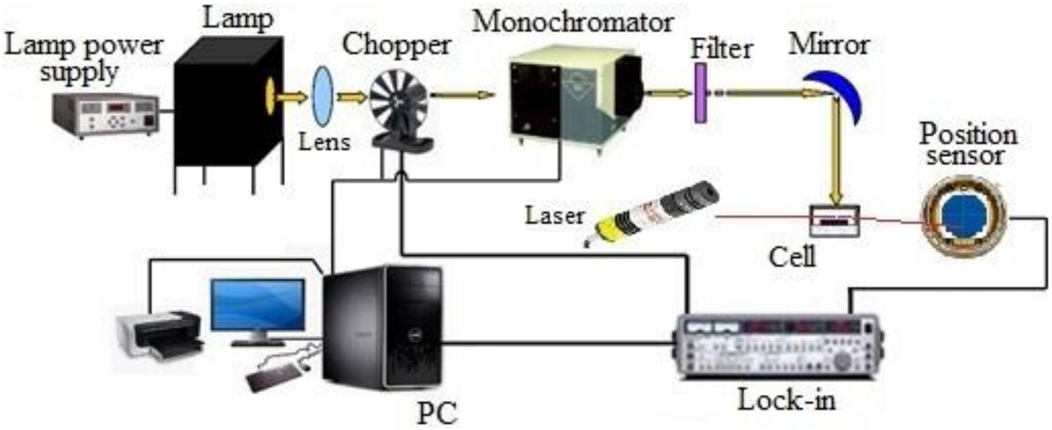


Figure 10. Photodeflection spectrometer

Photothermal imaging

The light source and the monochromator have been replaced by a laser source. The photoacoustic cell now rests on a XY positioning stage controlled by the PC. The scan step is very fine (5µm). Focusing optics replace now the elliptical mirror. It must provide a sharp focus to have a good resolution image. The motorized XY stage provides up to 13mm travel on both axes. Figure 11 depicts the photothermal imaging setup.

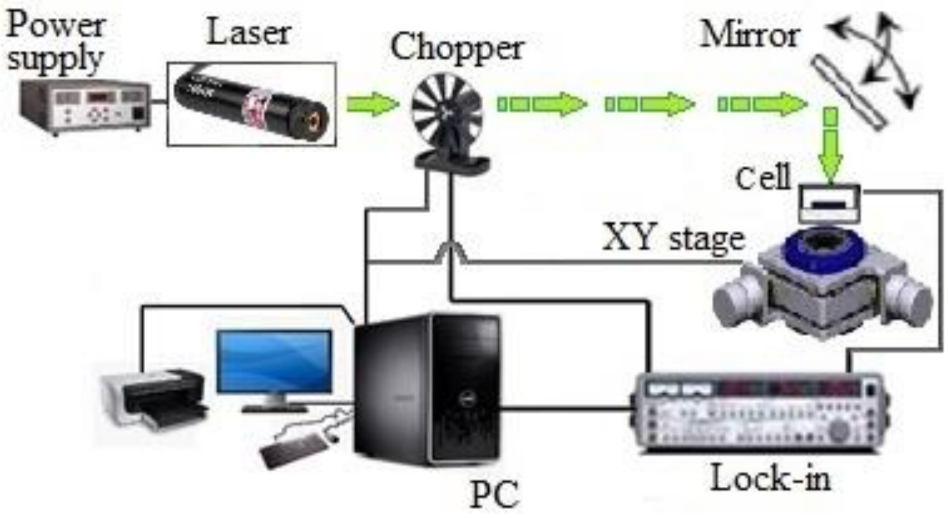


Figure 11. The photothermal imaging setup

Conclusion

To facilitate the characterization of new materials developed (in the field of photovoltaics and microelectronics) we have made available to researchers some powerful tools with very high sensitivity. The instrumentation of the three spectroscopy techniques is now assembled, it lacks only a few small accessories to make these techniques completely functional (focusing optics and mirrors). Once all the material is present one has to calibrate all three setups using reference or known samples.

References

Rosencwaig, A. (1980). *Photoacoustics and Photoacoustic spectroscopy*, New York, Wiley.
Sell, J.A. (1989). *Photothermal Investigations of Solids and Fluids*, Academic Press.
Zegadi, A. (1994). *Photoacoustic Study of CuInSe2 Single Crystals*, PhD thesis, University of Salford, UK.
Zegadi, A. Slifkin, M.A. Tomlinson, R.D. (1994). *Review of Scientific Instruments*, 65, 2238-2243.
Boccara, A.C. Fournier, A.D. Badoz, J. (1980). *Applied Physics Letters*, 36, 130-131.

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