

Applications of Steady-state Multichannel Spectroscopy in the Visible and NIR Spectral Region

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Abstract

Multichannel spectroscopy involves the detection of optical signals using linear or two-dimensional array detectors at the focal plane of a spectrograph. As there is no exit slit, there is an increase in throughput, as well as a direct multiplex advantage, making experiments both more rapid and sensitive compared to conventional single-channel techniques. Jobin Yvon's Optical Spectroscopy Division has a long history of providing world-class spectroscopic instrumentation, including spectrographs and multichannel detectors, to our customers. Recently, multichannel spectroscopy has opened up new possibilities in many areas of research, including materials characterization, plasma research, and biochemical analysis. In this article, we highlight some common applications of multichannel detectors of current interest in the scientific community.

1 Introduction

In recent years, spectroscopic techniques have been increasingly used in laboratories to characterize samples, for quality control or optimization of processes. Multichannel detectors have revolutionized spectroscopy by reducing the amount of time required to collect and analyze data, allowing researchers and engineers to monitor and make changes to their processes quickly, reducing time and wasted material.

A multichannel detection system offers two distinct advantages: the Fellgett (multiplex) and Jacquinot (throughput) advantage. Both of these advantages arise because, unlike a single-channel instrument, a multichannel detection system has no exit slit. One observes many wavelengths using many detection elements, simultaneously. This is the power of the technique – and it opens up possibilities for rapid data collection as well as kinetic data collection that are not achievable in any other manner.

2 Detector Installation into the Spectroscopic Instruments

2.1 Spectroscopic Instrumentation using Single-channel Detection

Spectroscopic measurement systems can vary from the simple to the complex depending on the actual information required. In its simplest form, a single-channel spectroscopy system contains a light source, which could be the sample itself (as in chemi- or bioluminescence), a sample holder, coupling optics, a spectrometer with a PMT or IR detector, and a data acquisition system. Light is gathered by sampling optics and passed into the spectrometer for analysis. The spectrometer disperses the beam into individual wavelengths using a diffraction grating. Rotation of the diffraction grating about its axis selects the wavelength of light that reaches the detector, positioned behind an exit slit. A full spectrum is collected by scanning the spectrometer across the wavelength range of interest. This can be a time intensive process, as each data point (wavelength step) may take a few seconds to collect, and each spectrum may take several minutes to complete.

2.2 Spectroscopic Instrumentation using Multichannel Detection

It is advantageous to increase data collection rates by utilizing multiple detection elements simultaneously. Polychromators achieve this by using multiple single-channel detectors placed at appropriate positions. Spectrographs differ from spectrometers and monochromators in that they focus the dispersed light across a focal plane (compared to a focal point or points). As such, they offer the possibility of continuous spectral detection across a wavelength range defined by the optical dispersion of the spectrograph with high resolution and throughput. Early instrumentation used photographic film positioned in the focal plane of a spectrograph to record such spectra.

Development of semiconductor charge-coupled device (CCD) detectors, such as Jobin Yvon (JY)'s Symphony family of CCDs (Fig. 1), has led to improvements in sample throughput and productivity in the lab. CCD detectors are silicon-based multichannel detectors that are divided into pixels. Each pixel, effectively an independent detector, records a signal proportional to the number of photons impinging upon it. Typical Symphony CCDs use 13 μm or 26 μm pixels, with array sizes of 512 to 2048 pixels long. This corresponds to a physical dimension of the focal plane that is between 12.8 mm and 26.6 mm wide.



Fig. 1 JY's Symphony CCD Family, including the Symphony Controller, an LN₂ Head and a Thermoelectrically Cooled CCD

A wide selection of arrays is available, depending on wavelength and resolution requirements.

When a Symphony CCD system is paired with a spectrograph, each pixel 'sees' a different wavelength allowing it to measure an entire spectrum at once. This allows an entire spectrum to be collected in a fraction of the time,

without having to move the grating and build a spectrum from a series of single-channel measurements. The Symphony CCD is excellent for signals in the UV, visible, and NIR wavelength ranges, from 200 nm to 800 nm.

In cases where the wavelength is greater than 800 nm, an InGaAs detector array is a perfect choice. The InGaAs Array was introduced to bring the speed and ease of acquisition of a CCD spectral acquisition to the NIR wavelength range. The InGaAs array uses a linear array of InGaAs photodiodes to collect spectral information.

2.3 Spectrometers and Spectrographs

Imaging spectrometers are commonly capable of using one array detector and one single channel detector such as a PMT or IR detector. A common arrangement uses a CCD for rapid measurements in the UV-VIS wavelength range (250 nm to 800 nm) as well as an InGaAs (indium-gallium-arsenide) single channel detector for the NIR wavelength range (800 nm to 1700 nm).

With the introduction of the TRIAX 322 and TRIAX 552 Dual Array Imaging Spectrographs, the opportunity to use two array detectors on the same spectrograph provides the advantage of multichannel spectroscopy over the full wavelength range. The TRIAX 322 and TRIAX 552 use the same optical arrangement as the TRIAX 320 and TRIAX 550, but provide a lateral array exit port in addition to the standard axial array port. The user may configure the spectrograph with any combination of two array detectors, as well as single-channel detectors, depending on their requirements. Fig. 2 shows the TRIAX 322.



Fig. 2 JY's Unique Dual Array Spectrometer System with a LN₂ Cooled CCD on the Front Port and a TE Cooled InGaAs Array on the Side Port

2.4 Sample Optics

A consideration that is sometimes overlooked when configuring a multichannel spectroscopic system is the sample optics. The sampling optics consist of optical elements (reflective, refractive, fiber-optic, etc.) that collect the light from the sample and pass it into the spectrometer. Consideration of the required wavelength range will determine the most appropriate materials and coatings. Matching of the optical aperture between stages is necessary to ensure proper performance, in terms of stray light, resolution and throughput, for the entire optical system.

3 Various Applications using the Multichannel Spectroscopy

Let's look at several examples of applications of multichannel spectroscopy that demonstrate the flexibility of the technique.

3.1 Photoluminescence

3.1.1 Photoluminescence Process in the Semiconductor Material

Photoluminescence spectroscopy (PL) is a powerful technique that is widely used to characterize semiconductors. It is non-destructive, and may be performed on a process line or in a quality control laboratory (off line).

In a semiconductor material, the electronic energy diagram may be described as having two bands as shown in Fig. 3. Electrons may be present in the ground state (valence band) or in an excited state (conduction band). The energy gap between the two bands is known as the bandgap. Measurement of this bandgap energy and its distribution are of importance in materials science, because they contain important information about the composition, quality, impurity levels and the energy level structure of the material.

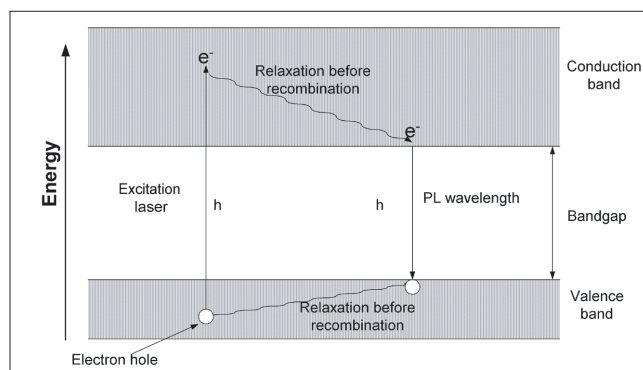


Fig. 3 Photoluminescence Energy Diagram

The incident laser light excites an electron from the valence band into the conduction band. Both the electron and electron hole relax so that the energy of the PL light is less than that of the excitation photon.

When light impinges on a sample, with energy greater than the bandgap energy, the light will be absorbed and an electron will be excited from the valence band, across the bandgap, and into the conduction band of the material. This excited state will decay, dropping across the bandgap from the conduction band into the valence band. When the electron makes the transition from the conduction band back down into the valence band, some of the energy given up by the electron is emitted as a photon that has the exact energy of the bandgap. This process is known as photoluminescence.

3.1.2 Experimental Results

Measurement of a photoluminescence emission spectrum allows rapid determination of bandgap energies. The emission wavelengths relate to the energy with the simple relationship: $\Delta E = h\nu = hc/\lambda$, in which c is the speed of light and h is Planck's constant.

In many cases, the materials scientist is interested in basic information about the bandgap energy and some limited information about the energy distribution. For such measurements, the sample is usually measured at room temperature, and the sample is being investigated in a macro sense, using a broad area. This type of setup often uses a fiber-optic probe or a system of lenses or mirrors to collect light from the sample. The light is then focused onto the entrance slit of the spectrometer for dispersion and measurement.

Fig. 4 shows Photoluminescence data from an Aluminum Gallium Arsenide and Fig. 5 photoluminescence data from an Indium Gallium Arsenide.

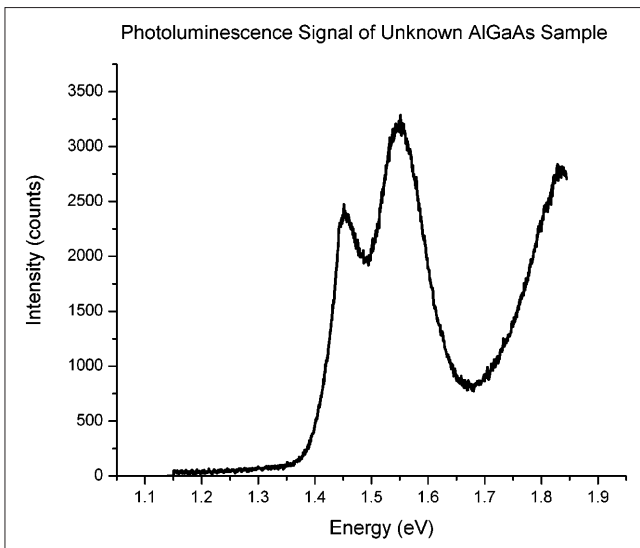


Fig. 4 Photoluminescence Data from an Aluminum Gallium Arsenide Sample Collected with a JY Liquid Nitrogen Cooled CCD

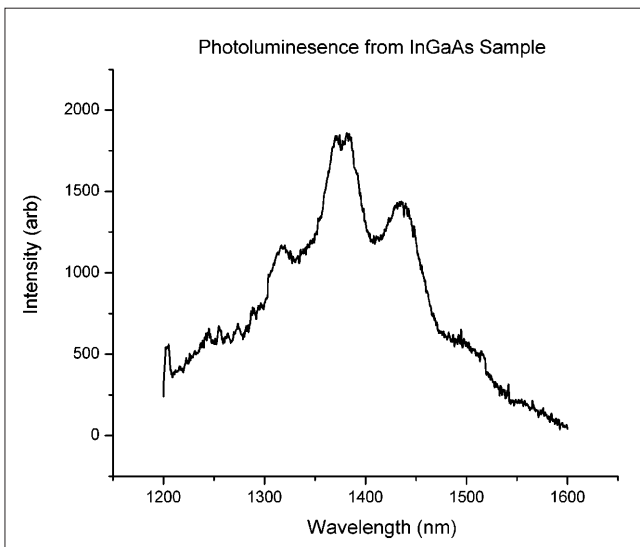


Fig. 5 Photoluminescence Data from an Indium Gallium Arsenide Sample Collected with a JY Liquid Nitrogen Cooled InGaAs Array

3.1.3 Applications

How does multichannel spectroscopy help? Photoluminescence spectroscopy has been actively studied for many years, using a scanning monochromator and photomultiplier tube (PMT). Such scans may be time-consuming, especially if the signal is weak and high-resolution spectra are required. Multichannel detection techniques, in the UV-VIS to the NIR, have allowed photoluminescence to be taken from the research lab and moved into process and quality control environments. Sample throughput is of importance in these settings, and multichannel detectors allow researchers to process more samples in less time.

3.2 Triboluminescence

Triboluminescence is emission of light triggered by mechanical energy from friction or, more commonly, fracture of a material. The exact reasons why particular materials show triboluminescence vary, but it is probable that crystal structure and impurities play important roles. Commonly known triboluminescent materials include wintergreen candy and quartz. Other minerals, such as calcite and mica, are known to exhibit triboluminescence when struck or rubbed.

One useful application of triboluminescence is in detecting impact damage of materials. Using triboluminescent compounds that glow when struck, researchers are attempting to make impact damage in composite materials easier to detect.

Triboluminescence emission may be generated by grinding the material under investigation in a sample holder, such as a simple test tube. This test tube is arranged, with appropriate coupling optics, at the entrance slit of a high throughput spectrograph and CCD system. Triboluminescence is generally weak, and the spectroscopic system should be fully optimized to give the best possible signal-to-noise ratio (S/N). A LN_2 -cooled CCD detector, with a high quantum efficiency back-illuminated CCD is ideal for this experiment. Back-illuminated CCDs offer high quantum efficiency (typically 90 % at 650 nm), so the sensitivity is enhanced.

The multichannel properties of the CCD detector have allowed triboluminescence spectroscopy to become a new tool in chemical analysis. As a CCD is an integrating detector, it properly accumulates the spectrum across the entire wavelength range, as the sample is being mechanically agitated. Previously, triboluminescence experiments were performed using a PMT and a scanning spectrometer. The low emission light levels, coupled with the difficulty of precisely grinding the sample for each data point, often meant long data collection time. A full spectrum of a material could take hours to complete. A CCD-based instrument is able to complete the spectral acquisition in a matter of minutes.

Fig. 6 shows triboluminescence emission of a sugar sample measured with a TRIAX 320.

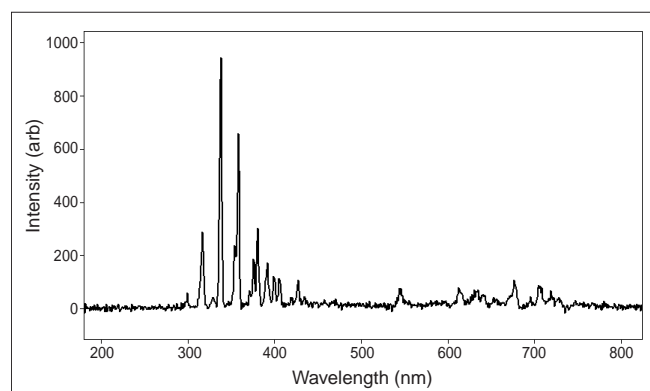


Fig. 6 Triboluminescence Emission of a Sugar Sample Measured with a TRIAX 320 and CCD System 3.3 Multitrack Spectroscopy

3.3 Multitrack Spectroscopy

CCDs are *area* detectors, and as such, possess the ability to collect information in 2 dimensions. When combined with an imaging spectrograph such as the TRIAX series, the horizontal axis of the CCD detector maps to spectral information. The vertical axis of the detector maps to a spatial position on the entrance slit. Typically, in spectroscopy, one adds all of the vertical pixels at a given horizontal position together, increasing the magnitude of the signal. This process is known as binning, and it effectively reduces the dimensionality of the CCD from two to one. With our SynerJY™ software, binning is flexible and easily defined in both horizontal and vertical directions.

A new application, known as multitrack spectroscopy developed from the combination of an imaging spectrograph and CCD. In this application, *multiple* optical signals are brought to the entrance slit of the spectrograph by multiple fibers. Each fiber represents a single signal, separated in the vertical direction, from the other fibers.

Now, by defining the whole area of the CCD as a set of areas, the instrument can read the signal from each fiber as an independent signal, at the same time. This mode of operation is useful when a reference spectrum and a sample spectrum are recorded simultaneously (as in absorption techniques), multiple sampling points in a process, multiple samples such as microwell plates, or any other application when the data must be collected in a simultaneous nature. Kinetic techniques benefit as well, because time-dependent changes may be collected and normalized in real-time, providing properly corrected data.

Fig. 7 shows a series of fibers at the entrance slit of a TRIAX spectrograph, being imaged onto the CCD area. Each signal is clearly resolved from its vertical neighbors.

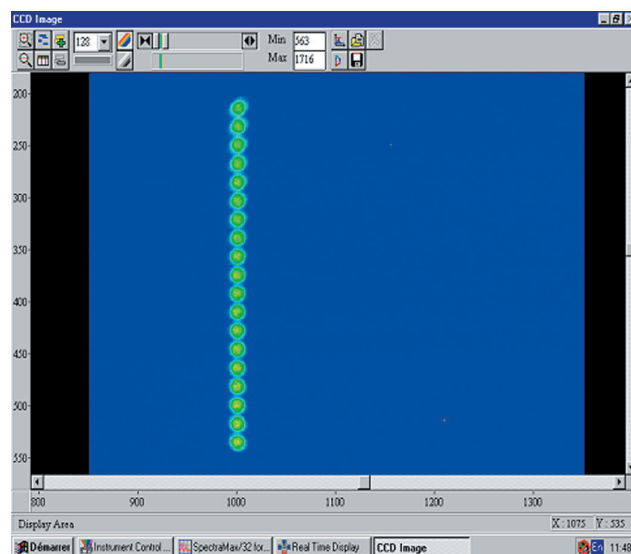


Fig. 7 CCD Image of a Fiber Bundle Collecting Data from a Mercury Lamp

The horizontal axis holds spectral information and the vertical axis contains positional information

4 Conclusion

Multichannel analysis methods, using linear and two-dimensional array detectors, open new possibilities for research. With the inherent advantages of multiplexing and throughput, multichannel detectors support new kinetic methods and more rapid spectral methods than accessible using single-channel techniques alone. The Optical Spectroscopy Division of JY specializes in producing top quality multichannel spectroscopic systems, based on Symphony CCDs and TRIAX spectrometers. We focus on our customer's needs and expectations, and our goal is to provide our customers with the best instrumentation and customer support.



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