

Multi-wavelength images detector for micro-cathodoluminescence analysis

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Recibido el 23 de marzo de 2006; aceptado el 8 de junio de 2006

A novel instrument capable of collecting the entire cathodoluminescence emission spectrum simultaneously with SEM scanning is presented in this work. The cathodoluminescence produced in a standard scanning electron microscope is collected and analyzed. The electronic signal used by the microscope to trigger the electron raster and produce an image is also used by the spectrograph to synchronize spectrum acquisition. At each point in the sample the complete spectrum is acquired. All the data are recorded and saved in an electronic file. With this information it is possible to reconstruct panchromatic and/or monochromatic images of the sample and correlate its optical properties with its microstructure. Instead of an elliptical mirror to collect the light in the conventional CL system, a single optical fiber is used. This new way of acquiring light permits a reduction in the work distance (down to 5 mm) in the microscope, thus achieving a better spatial resolution. Another advantage of this arrangement is that we can simultaneously use other detection modes such as backscattered electrons, x-rays etc. Examples showed here illustrate the capabilities of the instrument.

Keywords: Cathodoluminescence; spectroscopy.

En este trabajo presentamos un novedoso instrumento capaz de coleccionar el espectro de emisión completo de cátodoluminiscencia, simultáneamente con imágenes de SEM. Se colecciona y analiza la cátodoluminiscencia producida en un microscopio electrónico de barrido. La electrónica utilizada por el microscopio para barrer el haz de electrones y producir la imagen es utilizada por el espectrógrafo para sincronizar la adquisición de espectros. En cada punto de la muestra se adquiere el espectro completo. Todos los datos son grabados y guardados en un archivo electrónico. Con esta información es posible reconstruir imágenes pancromáticas y/o monocromáticas de la muestra y correlacionar sus propiedades ópticas con la microestructura. En lugar de un espejo elíptico para coleccionar la luz, como en el sistema convencional de CL, se utiliza una sola fibra óptica. Esta nueva manera de adquirir la luz permite una reducción en la distancia de trabajo (menor a 5 mm) en el microscopio, logrando así una mejor resolución espacial. Otra ventaja de este arreglo es que nosotros podemos usar otros modos de detección simultáneamente como los electrones retrodispersados, rayos X, etc. Se muestran algunos ejemplos para ilustrar las capacidades del instrumento.

Descriptores: Catodoluminiscencia; espectroscopia.

PACS: 78.60.Hf; 07.60.Rd

1. Introduction

Characterization of microscopic properties in material science has reached maturity, particularly with electron spectroscopies, both in image and analysis modes. Cathodoluminescence (CL) mode of an electron probe has been widely used in laboratories since 1980s in the assessment of optical and electronic properties of solids. This technique provides information on impurity levels, and an analysis of dopant concentration at a level that is several orders of magnitude lower than that attainable by other techniques. Mapping defects and determination of carrier lifetimes and charge carrier capture in a cross section of impurities can also be obtained using CL (Holt 1981).

Cathodoluminescence has been extensively used in modern cathode-ray tube (CRT)-based instruments. With the development of electron microscopy techniques, CL has emerged as an important characterization tool for the analysis of luminescent materials. SEM and TEM with a CL attachment are well suited for the microcharacterization of materials, because they provide high spatial resolution and a

simultaneous variety of acquisition modes of data. CL analysis in a SEM is a unique technique because it permits the microcharacterization of the optical and electronic properties of luminescent materials in the same instrument. A CL analysis can be performed in imaging or spectroscopy mode.

The choice of a detector is very important in CL measurements. Photomultipliers are the most efficient detectors in the visible range, and recently an increasing use of solid state devices and Fourier transforms spectrometers for detecting luminescence in the visible and infrared range has been reported. Koschek and Kubaleck (1985) described the characteristics of various detectors used for CL measurements. The essential requirements for the design of a CL detection system are a high efficiency in light collection, transmission, and detection. Usually light collection is performed locating the sample at one focus of an elliptical mirror and capturing the light with an optical fiber at the other focus. Nevertheless, the elliptical mirror limits the working distance of the microscope, thus reducing the spatial resolution. The collected light is transmitted by the optical fiber into the spectrometer through an entrance slit. The spectral resolution is improved

by reducing the width of the entrance slit, but the light intensity coming to the spectrometer is also strongly reduced.

Earlier cathodoluminescence detector designs had been reported since the 1980s by Holt (1981) and Trigg (1985). They collected light using light guides or ellipsoidal mirrors directly coupled to a monochromator. A photomultiplier captures the monochromatic light, which in general is a weak signal. Steyn *et al.* (1976), reported for the first time the use of a fiber bundle in combination with a lens and ellipsoidal mirror. Another innovation is the use of an intensified charge couple device (ICCD), which is more sensitive than the conventional CCD.

In this paper we report a novel instrumentation for capturing CL spectral images in a SEM. With this instrument it is possible to obtain all the spectral information of a sample in a single experiment, thus reducing the damage caused by exposition to electrons. The dwell or exposition time (10 to 100 milliseconds) and the signal to noise ratio are similar to that reported in the literature (Contreras *et al.* 2002). In our setup, the working distance of the SEM is smaller than that attained with the use of an elliptical mirror. We present some examples showing the capabilities of this instrument.

2. Experimental setup

We adapted a CL instrument to a standard SEM, Jeol 5300. An optical fiber is introduced into the specimen chamber of the SEM through a SMA connector. One end of the optical fiber, inside of the specimen chamber, is brought close to the sample. The other end goes to the CL setup. A schematic diagram of the CL instrument is illustrated in Fig. 1.

Light collection is carried out by an optical fiber 1.5 mm in diameter located at 4 mm from the sample at an angle of 45 degrees. The collected light is transmitted by the optical fiber and coupled to the monochromator through a fiber bundle. The fiber bundle was made in one end, with a bundle of fibers in a circular arrangement, packed together by mechanical pressure. In the other end of the fiber bundle, the individual fibers are aligned in a straight line (see Fig. 2).

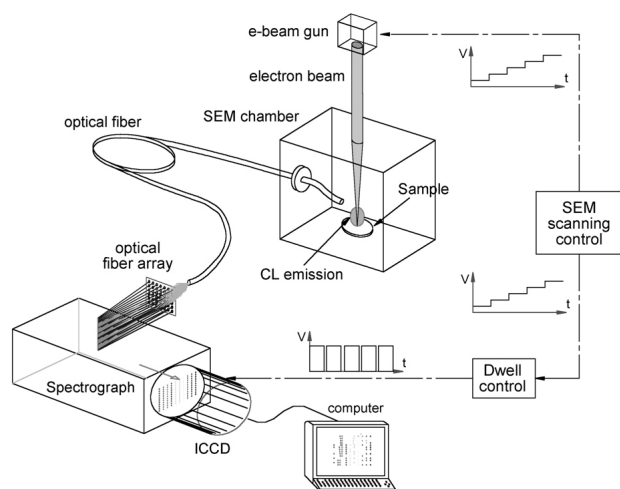


FIGURE 1. CL-SEM setup.

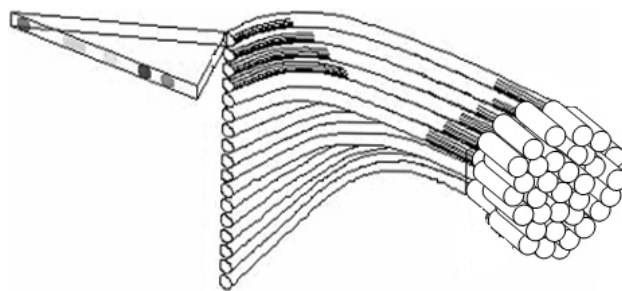


FIGURE 2. Bundle fiber array.

This linear array of fibers plays the role of monochromator entrance slit. The area in both extremes of the fiber bundle is the same; in one case it is rectangular, in the other a circle. The dimensions of the circular and linear array of fibers are related by the following equation:

$$4HW = \pi D_f^2, \quad (1)$$

where H and W are the height and minimum width of the monochromator slit, and D_f is the fiber bundle diameter. The bundle fiber was fabricated using fibers of 0.250 mm (W) in diameter. The height of the linear array was set to 7 mm (H), which gives a size of the circular arrangement of 1.5 mm (D_f) in diameter. With this accessory, all the collected light is analyzed by the spectrometer. A Czerny-Turner type monochromator was designed with $f/N=8$ interchangeable gratings. The gratings are mounted on a metallic plate with kinematic support, to change them. With a 300 l/mm grating, the 190-850 nm range is divided into two parts, overlapping 10% to match the graphics; every section has a bandwidth of 300 nm, corresponding to 0.74 nm/pixel. Our CCD has an intensifier, 256×512 pixels, 20 micrometers square in size, with a minimum gate of 2 ns, air and water-cooled (R. Machorro *et al.* 2002). The electronic signal of the SEM is used by the ICCD in order to synchronize the e-beam position with the spectra. This task is accomplished with an electronic device to transfer a step function into a pulse train. The electron beam moves each voltage step, and the pulse drives the ICCD shutter.

At every point, once the electron beam impinges on a sample region, the spectrum is obtained and saved digitally as well as the secondary electron image.

At the end of the raster beam, and SEM image is completed, as well as the spectra at the same number of points.

A multi-wavelength CL experiment of the sample is performed only once. All spatial and spectral information is stored in an electronic file. The formation of wavelength images are obtained by extracting from the file the intensity corresponding to the desired wavelength and position.

3. Results and discussion

Figure 3a shows the morphology of ZnS:Cu particles as observed by low magnification SEM imaging. The image taken with $\lambda = 425$ nm (Fig. 3b) shows uniform emission from the

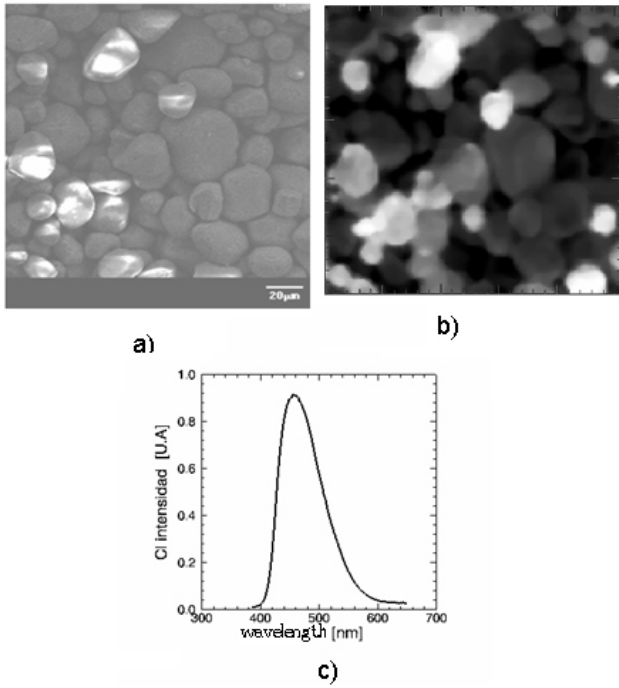


FIGURE 3. CL Image and spectra 350 to 600 nm of ZnS sample.

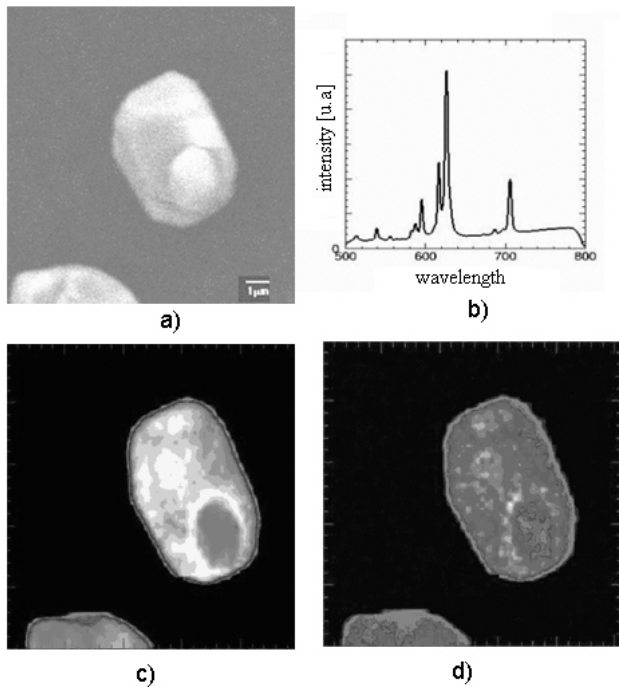


FIGURE 4. CL Image and spectra 350 to 600 nm of Y2O3:Eu sample.

whole particles. No additional features are observed from the surface particles.

The spectral emission of the ZnS powder is depicted in Fig. 3c. The ZnS particles have an asymmetric, broad emission with a maximum emission in the blue range, around 425 nm.

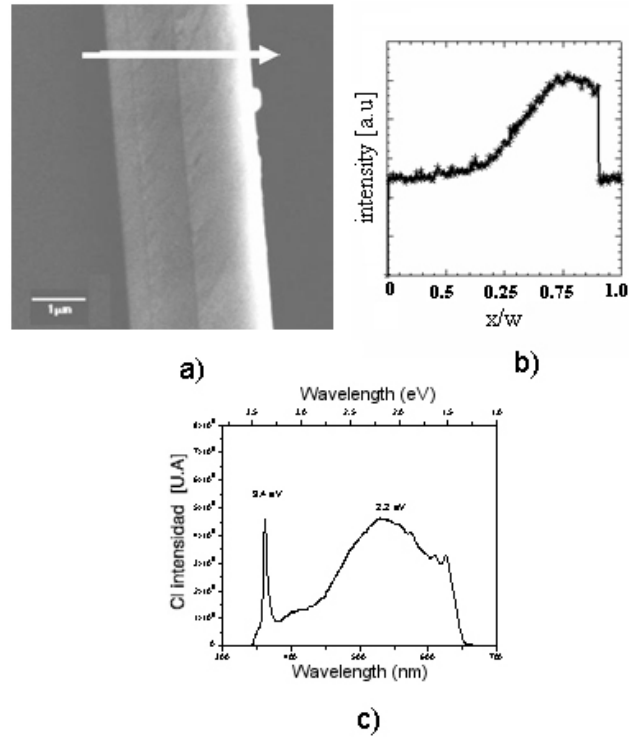


FIGURE 5. SEM Image of GaN:Si sample. The cross-section intensity profile obtained by scanning the 362 nm emission of GaN across the film.

A 5 μm single particle of Eu doped Y₂O₃ is imaged by SEM in Fig. 4a. The image illustrates a very small aggregate above 2 μm that seems coalesced during the growth. The spectrum (Fig. 4d) shows the typical sharp emissions of the Eu³⁺, corresponding to the atomic transitions of the ion. Two monochromatic images at 611 nm and 714 nm are shown in Figs. 4b and c, respectively.

The instrument also makes it possible to obtain intensity profiles from cross-section samples, as illustrated in Fig 5.

A heterostructure of GaN layers grown on Si(111) observed in cross-section is shown in Fig. 5a. The intensity profile shown in Fig. 5b was obtained by scanning the 362 nm emission of GaN across the film following the line depicted in the SEM image. An entire spectrum of the sample shows the band-edge emission of the GaN in addition to the broad yellow emission that in the literature is correlated with crystal defects and impurities (S.O. Kucheyvey *et al.* 2002) [Fig 5c].

4. Conclusions

We reported an instrument able to obtain wavelength images from luminescent samples. The spatial resolution of the instrument was improved by substituting the elliptical mirror with an optical fiber. The use of intensified CCD allows for a good signal to noise ratio, thus reducing the exposure time to values in the range of 10 -100 msec. The introduction of a bundle fiber array makes it possible to improve the spectral resolution without losing to much light, as happens with the convention slits.

Acknowledgments

The authors' thanks to M. Farfán, G. Vilchis, A. Tiznado, Enrique and J. Peralta for their technical assistance. This work

was done in part with support from Conacyt-México, under contract 400380-5-G36531-E. We also wish to thank to Durel Corporation for providing the ZnS powder.

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