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Use of a silicon drift detector for cathodoluminescence detection

M. Béranger *

Trixell, 460 rue du Pommarin, BP no. 21, 38346 Moirans, France

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ABSTRACT

Cathodoluminescence, the study of light emission of a sample under the electron beam of a scanning electron microscope, is an efficient technique for luminescent material characterization and for defect analysis in semiconductors. However, the purchase of full cathodoluminescence equipment is not always possible for economic reasons. This study shows that it is possible to use a silicon drift detector to detect visible light, instead of buying a cathodoluminescence system. First, the SDD response is characterized with a light illuminator and then the behaviour is confirmed with a scintillating material. When the excitation level is kept low enough, the response of the silicon drift detector to light is an intense peak at low energy in the X-ray spectrum. Several scintillating materials with different sensitivities are used to show that the variation of the count rate of the low energy peak with the excitation level is related to the quantity of emitted light. This method allows the characterization of the homogeneity of a luminescent sample.

1. Introduction

Cathodoluminescence (CL) is the study of light generated in semiconductors or insulating materials under electron excitation [1]. First performed in the field of mineralogy and in studies of defects in semiconductors, this method becomes increasingly used with the development of optoelectronics devices.

In some cases, only the light intensity is needed. The most common attachment in a scanning electron microscope (SEM) is not the CL equipment but the silicon drift detector (SDD) for energy dispersive X-ray spectrometry (EDX). Compared to conventional Si(Li) detectors, SDDs offer a superior count rate capability associated with a high resolution and light weight, without the need of liquid nitrogen cooling [2–5]. The SDD is sensitive to light, as shown by its use as γ-ray detectors [6,7] and this sensitivity can be used to perform panchromatic CL. Few studies have been published on this subject. This method was demonstrated by P.F. Smet et al. [8] on $BaAl_2S_4:Eu^+$ films.

The aim of this work is to characterize the response of a SDD to visible light and to find the operating conditions allowing its use for panchromatic luminescence. After tests with visible light, a scintillator material is observed in the SEM using the SDD as light detector. A way to quantify the local light emission of a sample is proposed.

* Corresponding author.
E-mail address: Murielle.beranger@trixell-thalesgroup.com.

2. Equipment and materials

The SEM used in this study is a Leo 440 equipped with a tungsten filament. Measurements are conducted with an Inca X-Act SDD system, from the supplier Oxford, with a 10 mm^2 chip. Except when specified otherwise, acquisition time is set to 60 s, voltage to 20 kV, working distance to 23 mm; the electron beam impacts an area of 50 × 50 μm. A new area of the sample is used for each measurement. To homogenize operating conditions from day to day, before each set of measurements the SEM filament and column are settled on the cobalt standard to get about the same number of counts in the L-peak of cobalt.

Two types of scintillating material used in X-ray detectors are tested in this study: cesium iodide doped with thallium, CsI:Tl or gadolinium oxysulfide, Gadox [9,10]. CsI:Tl is made at Trixell by evaporation on a thin aluminium substrate. Gadox is obtained from the supplier Kyokko.

The scintillating material is polished in order to get a flat surface and avoid shadowing effects due to surface reliefs. As it is an insulating material, it is discharged with copper adhesive and by the evaporation of a thin layer of carbon on its surface. Two samples were used for each carbon evaporation: a flat sample prepared for SEM observation and another sample from which the luminescence was measured before and after carbon evaporation. The ratio between the quantity of light measured after carbon evaporation and the one measured before gives access to the absorption of the carbon layer. Luminescence is measured under X-ray excitation at 70 kV. Light spectrum is obtained by the use of a monochromatic system coupled to a charge coupled device image sensor.

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For experiments with external light, the light illuminator is a halogen light fibre model EK1 from the supplier Euromex microscopes Holland.

3. SDD response to light

3.1. Tests with a light illuminator

First, the behaviour of the SDD under visible light is checked using a light illuminator. During these tests, the SEM chamber is under vacuum, the electron beam is off and the inside of the SEM is illuminated through the chamber window by a light illuminator while the EDX signal is collected (see Fig. 1, where number 1 represents the SEM window and 2 is for the sample holder and stage).

Fig. 2 displays a few spectra measured on the light illuminator for different excitation levels. The quantity of emitted light is calculated from these spectra as a function of the illumination level. During the SDD illumination tests with this light illuminator, only a low energy peak is observed on the EDX spectrum. The height of this peak changes with light intensity. Following ref [8], this signal is representative of the visible light. To characterize the variation of the low energy peak with light intensity, different parameters can be calculated from the EDX spectrum and from information given by the software [5,11]:

- The throughput count rate is the count rate of X-ray or light pulses which are successfully processed by the detector electronics, and it corresponds to the number of counts in the spectrum per unit of total counting time
- The input count rate is the number of X-ray (or light) pulses per second that are sent to the detector electronics, no matter if they are processed or rejected
- The dead time is the time associated to pulses rejected by the detector electronics, usually expressed as a percentage of the total counting time.

The throughput count rate due to this low energy peak is shown on Fig. 3 as a function of the quantity of emitted light. The variation of the input count rate with light follows the one of the throughput count rate. The evolution of the dead time is the same as the one of the count rate with illumination. In the low intensity domain, the response of the SDD increases with the quantity of light and this domain should be chosen to use the SDD as a light detector.

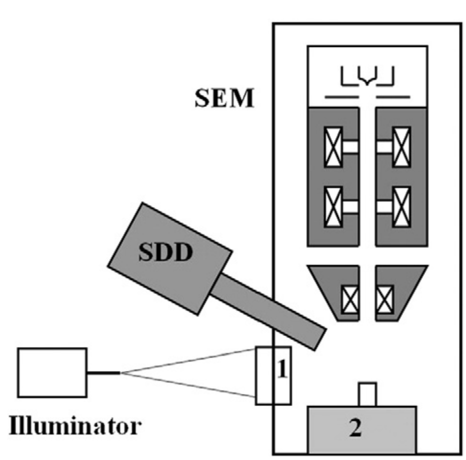

Fig. 1. Experimental set-up.

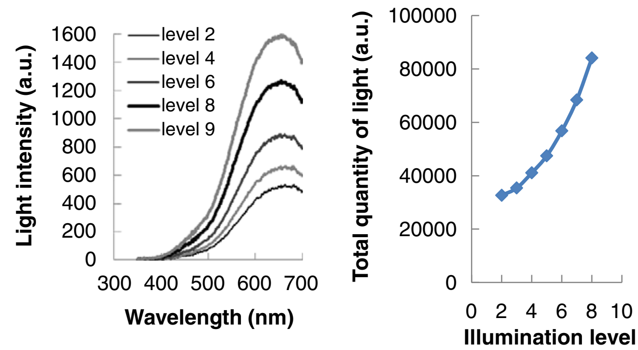

Fig. 2. Light illuminator spectra and intensity.

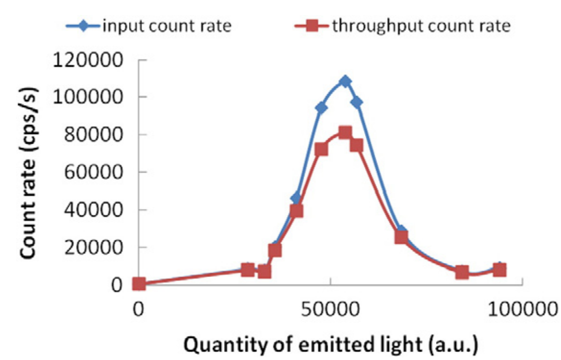

Fig. 3. Variation of throughput and input count rates.

3.2. Tests with scintillators

A CsI:Tl sample is used to test the capability of the SDD to detect visible light. This material grows in the form of needles and was cut along the needles axis (as shown in Fig. 4). Under excitation, CsI:Tl emits visible light with wavelength between 350 and 750 nm, the maximum being located at about 550 nm, which corresponds to green light [9].

The EDX spectrum obtained on a CsI:Tl sample shows the Cesium and Iodide L peaks, and a high signal is observed on the low energy channels (Fig. 5). This signal is much higher than the one measured on a non-luminescent Co standard in the same conditions and is representative of the visible light emitted by the sample.

The throughput and input count rates in the low energy peak (E < 0.2 keV) are shown in Fig. 6 as a function of probe current for the luminescent CsI:Tl material. When probe current increases, the throughput and the input count rates increase very rapidly, reach a maximum, decrease and stabilize at a low value. This behaviour is similar to the one observed with the visible light from the illuminator, but

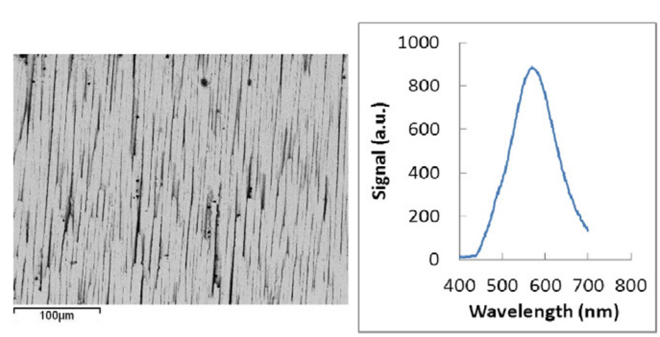

Fig. 4. CsI needles and light spectrum.