

LabVIEW-based control and data acquisition system for cathodoluminescence experiments

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(Received 28 June 2011; accepted 28 October 2011; published online 30 November 2011)

Computer automation of cathodoluminescence (CL) experiments using equipment developed in our laboratory is described. The equipment provides various experiments for CL efficiency, CL spectra, and CL time response studies. The automation was realized utilizing the graphical programming environment LabVIEW. The developed application software with procedures for equipment control and data acquisition during various CL experiments is presented. As the measured CL data are distorted by technical limitations of the equipment, such as equipment spectral sensitivity and time response, data correction algorithms were incorporated into the procedures. Some examples of measured data corrections are presented. © 2011 American Institute of Physics. [doi:10.1063/1.3662203]

I. INTRODUCTION

Cathodoluminescence (CL) is an opto-electronic phenomenon occurring in a large number of non-metallic materials when a light emission is created after irradiation by an electron beam. Intensity, spectral distribution, and time response of the CL emission are characteristic parameters for every material. Therefore, this phenomenon is widely used for material imaging and microcharacterization in electron microscopy.¹ CL also provides important informations for the development of many electronic components, such as cathode ray tube fluorescent screens² or electron scintillation detectors.^{3,4}

There are several commercial systems for CL measurement on the market. These devices typically operate in combination with light microscopes or scanning electron microscopes.⁵ Most of these systems are primarily focused on CL imaging and spectroscopy; however, they do not usually provide time response studies of the CL emission. In addition, the price of these systems is mostly high. For these reasons, specialized CL equipment was developed in our laboratory in recent years.³ This experimental instrument meets the high variability of CL studies, efficient CL emission collection, and reasonable price. The equipment has already brought remarkable results.^{6–8}

CL experiments performed by the mentioned equipment can be divided into two groups differing in e-beam excitation of a specimen. (1) Continuous excitation means that the specimen is continually excited and the generated CL emission is studied, for example, as a function of time, emission wavelength, specimen temperature, or of e-beam current density. This is useful for the study of CL properties, such as efficiency, material degradation, and emission spectra.^{9,10} (2) Pulsed excitation means that the specimen is excited by a modulated e-beam. In this excitation method, the time response of the CL emission is studied in dependence on various parameters, such as emission wavelength, specimen

temperature, or excitation pulse length. This method is useful to describe electron kinetic properties in materials.^{11–13}

During the experiments, it is necessary to control a number of individual instruments, monitor parameters, and record experimental data. Therefore, satisfactory results cannot be obtained without automation of the experiment. Our previous approach was characterized by an effort to write software for automatic control and data acquisition using a text-oriented programming language. Unfortunately, this solution possessed disadvantages in long development stages and in difficult maintenance and modification of the software. Therefore, we chose an approach using the graphical programming platform LabVIEW developed by National Instrument.¹⁴ It was already proved that LabVIEW is an excellent tool for experiment automation.¹⁵ In the last two decades, LabVIEW found wide applications in instrumentation in various fields, especially in physics, such as optics,^{16–18} thermodynamics,^{19,20} plasma physics,^{21,22} and microscopy.^{23,24}

Compared with a text-based programming, the graphical programming is characterized by the simplicity of application software development and relatively easy readability of source code. Moreover, LabVIEW is mainly focused on instrument control.²⁵ It offers extensive support for communication with scientific instruments and their control by a computer. Drivers for many different types of instruments and buses are included in LabVIEW installation or are available from instrument producers. In addition, LabVIEW contains many libraries with a large number of functions for different fields, such as data acquisition, mathematics, signal generation, or analysis. These additional libraries effectively shorten the development time.

II. CL EQUIPMENT SETUP

CL studies were performed using the equipment built in our laboratory and shown in the schematic diagram in Fig. 1. A specimen irradiated by an electron beam (5–20 kV) is placed into a vacuum chamber. The e-beam focused by

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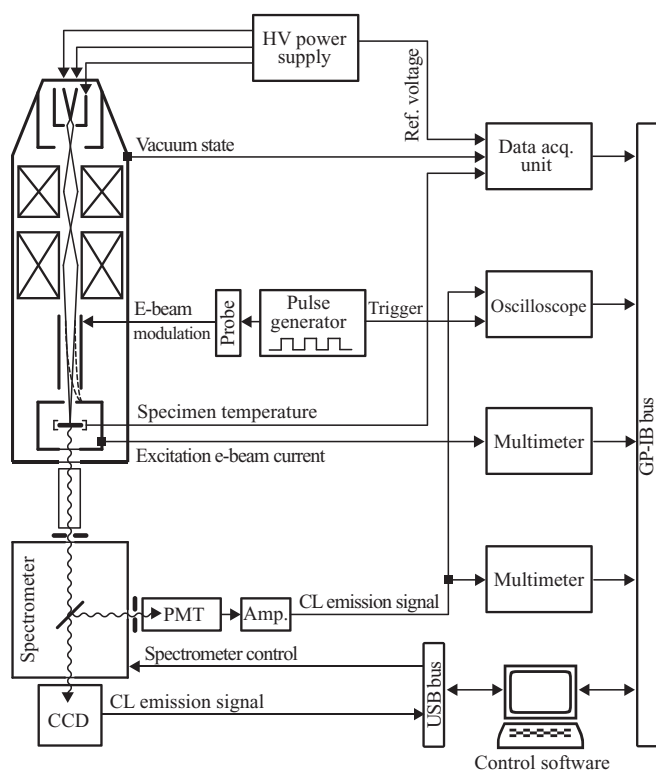


FIG. 1. Schematic diagram of CL equipment instruments and their signal connections.

electrostatic lenses is generated by thermal-emission cathode. It can be modulated by an electrostatic deflection system located above the specimen. This system is able to produce e-beam pulses of variable width (minimum is 50 ns) with 800 ps rise and fall time (measured in the range 10%–90% of the intensity). The e-beam modulation is necessary for the pulse excitation of the specimen. To enable e-beam current measurement, a Faraday cage is located around the specimen. The measured current is converted to the voltage and sampled by the Agilent 34401A multimeter. The specimen chamber is equipped with an electric heater for specimen heating to 550 K, which is used for *in situ* temperature dependence experiments. The CL light emission is collected directly to a silica light guide from the specimen side opposite from the e-beam irradiation. This realization has a big advantage of efficient light collection; however, the specimen has to be either optically transparent, or in the form of a thin layer on an optically transparent substrate.

The CL emission is guided directly to a light detector or to a slit of the spectrometer Horiba JY iHR320. The theoretical spectral resolution of the spectrometer used is 0.1 nm; however, due to usual low intensities of light, the CL experiments are carried out with the slit width about 1 mm and the spectral resolution about 3 nm. Two detectors with wide spectral sensitivity (200–900 nm), such as the photomultiplier tube (PMT) Hamamatsu R943-02 and the CCD camera Horiba JY Synapse, can be placed at the output ports of the spectrometer. Detector choice is performed by flipping the mirror inside the spectrometer. Spectral measurements using PMT are slower but more sensitive in comparison with the CCD camera. Moreover, PMT has usually faster time response; the

rise time of PMT used is 3 ns (10%–90%). This paper deals only with the alternative of using PMT. The electric current from PMT is amplified and converted to a voltage signal by a fast operational amplifier, and then sampled by the Agilent 34401A multimeter. Signal noise is reduced by mathematical integration over an appropriate time interval. At the pulsed excitation, a periodically varying signal is recorded using the 400 MHz oscilloscope HP 54504A. It is also important to measure various experimental parameters, such as specimen temperature, e-beam energy, or vacuum state. These values are converted to electrical signals and sampled using the data acquisition unit Agilent 34970A.

PC communication with each measuring instrument of the CL equipment is provided via the universal serial bus (USB) or the general purpose interface bus (GPIB) interface. Fast USB 2.0 is used for spectrometer control and data acquisition from the CCD camera. Other mentioned instruments do not require fast and large data transfer, so the slower GPIB interface is satisfactory.

III. EXPERIMENT AUTOMATION

The computer automation of CL measurements follows designed procedures, which are briefly described in this section utilizing the flowchart in Fig. 2. The procedures can be divided into three groups according to the type of experiment: (1) CL efficiency procedure, (2) CL spectrum procedure, and (3) CL time response procedure. In the CL efficiency experiment procedure (the middle branch in Fig. 2), the specimen is continually excited by the e-beam and the CL emission is observed especially not only in the dependence on time, but also in the dependence on excitation e-beam current or on any of the measured parameters, such as specimen temperature. The CL emission signal and the e-beam current are sampled using the two multimeters, and the mentioned parameters are sampled by the data acquisition unit. The simultaneous measuring of multiple signals and data acquisition via the GPIB is automatically performed by the LabVIEW application software.

The CL spectrum experiment procedure (the left branch in Fig. 2) is designed for CL emission recording as a function of the UV-VIS light wavelength. During the spectral procedure, it is necessary to control the spectrometer with the application software and to sample the CL emission signal, the excitation current, and the experimental parameters. Before the measurement, the spectrometer, multimeters, and data acquisition unit are configured. The main spectrometer setting is selection of the diffraction grating, the slit widths, and the length of the scan step. The scan step, used for setting of scanning spectra resolution, should always be considered carefully, since the spectral resolution is mainly limited by slit widths. Spectral scanning procedure begins with the initialization of the instruments. The application then scans step-by-step the individual wavelengths from the initial to the final wavelength position and immediately acquires measured signal between each step. The spectral scanning usually takes time, during which the intensity of the CL emission can decrease, for example, due to material degradation by the

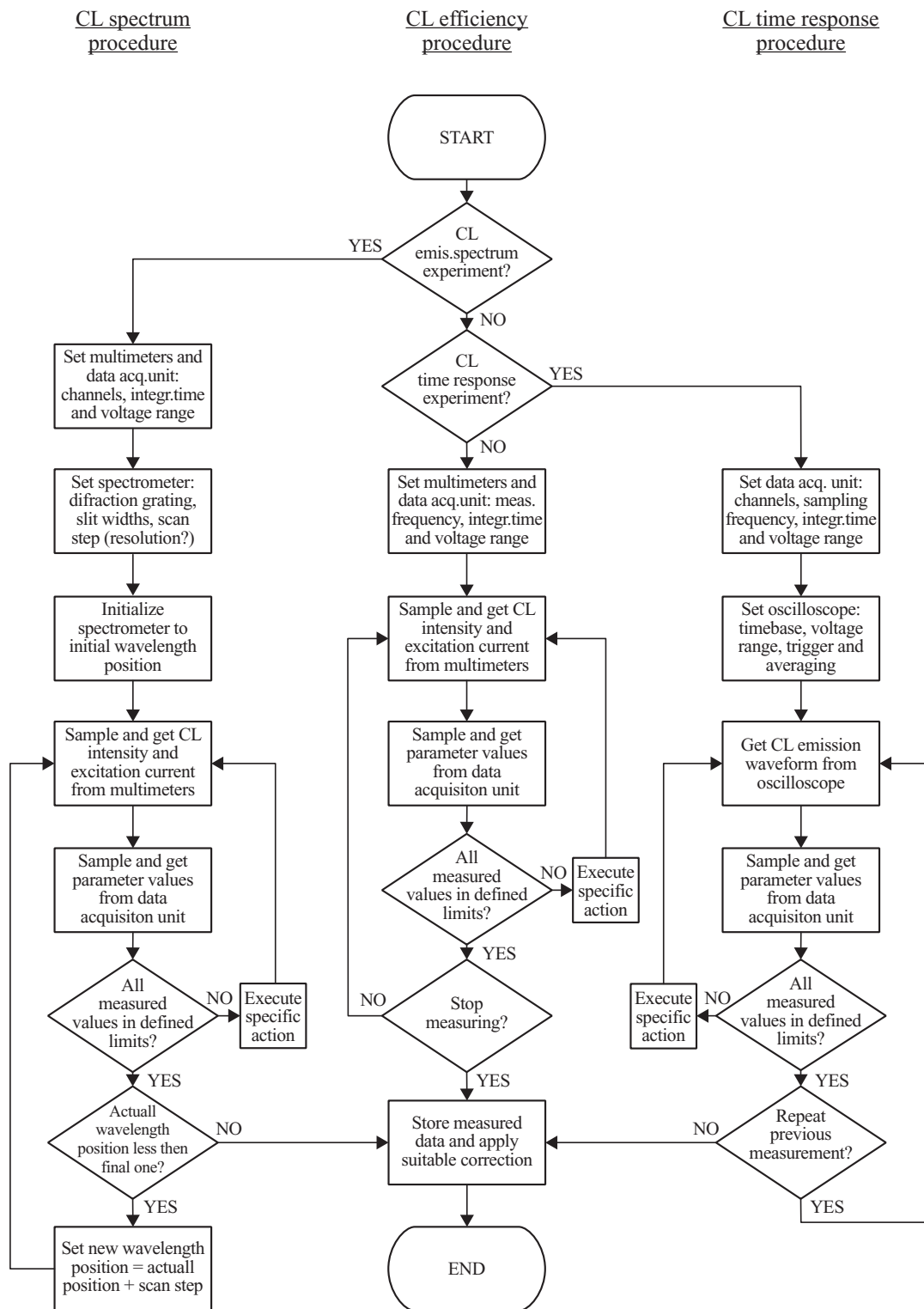


FIG. 2. Procedure flowchart of the CL experiments.

e-beam. Therefore, it is useful to scan a spectrum forward and backward for both measured spectra comparison.

The CL time response experiment procedure (the right branch in Fig. 2) is realized during the pulse excitation of the specimen, when the excitation e-beam is modulated. Dependence of the CL emission on time is then recorded by the oscilloscope. Before the procedure is initiated, the operator defines the oscilloscope setting using the LabVIEW applica-

tion. The next step is background noise subtraction, which is important for precise CL decay curves interpretation, especially in logarithmic scale. This process is done by shutting of the excitation e-beam and background signal measuring and storing as a signal offset. After that, the pulsed e-beam modulation is set and the application captures the shape of the CL emission curve from the oscilloscope and subtracts the offset. During the measurement, the values of excitation current

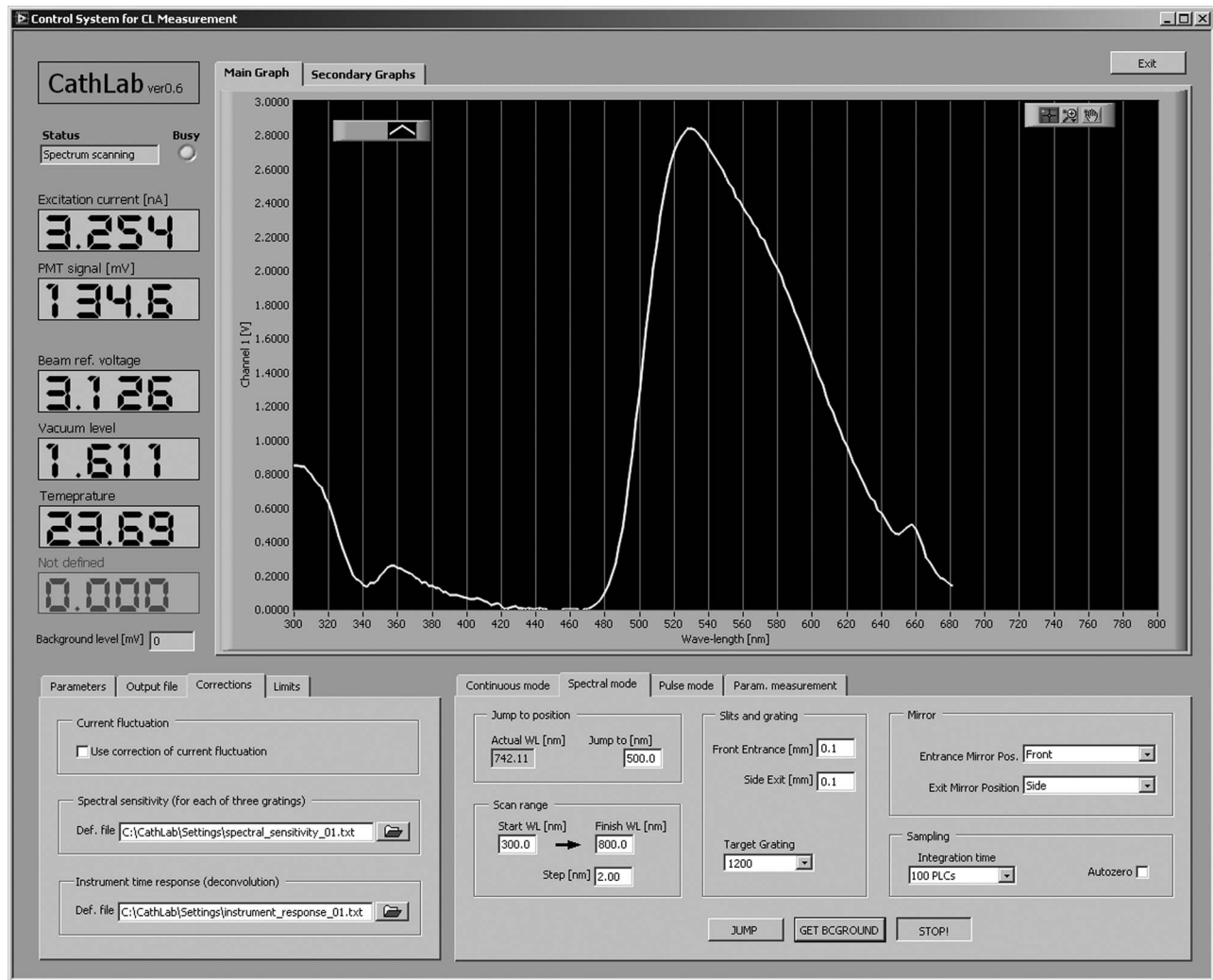


FIG. 3. The application software MS Windows GUI for the CL experiment control and for data acquisition and processing. The actual measurement of the CL emission spectrum of the YAG:Ce single crystal scintillator is shown.

and the experimental parameters are acquired. The recording of the CL emission curve can be repeated in loops, which is suitable for signal averaging and noise reduction.

It is necessary to monitor that some signals do not exceed allowed limits during the experiment. For example, excessive CL emission intensity could damage PMT, or a high excitation current could lead to specimen destruction. For this reason, the application checks the values during the measurement. If defined limits are exceeded, the application promptly executes a specific action, for example, a command sent to the power supply to reduce PMT voltage. If any danger emerges, the application generates at least a signal to the operator.

IV. LabVIEW APPLICATION SOFTWARE

The application software, including the described procedures for the CL experiments, was programmed in LabVIEW and it was named CathLab (Cathodoluminescence Laboratory). LabVIEW drivers from instrument manufacturers were used for software development. An application user interface was based on an intuitive control environment using a single

application window. Such an interface has a great advantage because the operator can control all of the instruments of the CL equipment in one application window.

A dominant main graph is displayed in the user friendly window as shown in Fig. 3, which clearly shows measured CL emission intensity as a function according to the selected experimental procedure, such as the function of time or wavelength. The operator can use tabs to switch the view from the main graph to the secondary graphs, which plot the values of experimental parameters. Numerical indicators on the left side of the window show the actual measured quantities, which are useful for the immediate measurement monitoring. In the bottom left part of the window, the tabs for output operation settings are situated. Here, it is possible to define output operations, such as an output text file, corrections applied to the measured data, and monitored limit values of measured signals. The bottom right part of the window contains tabs for the settings of the instruments and the experimental control. The control of each type of CL experiment according to the three individual procedures is located on a different tab. The fourth tab is used for setting the experimental parameter measurement.

Before the experiment is started, the operator first sets the parameters of output operations, then selects the appropriate tab for the procedure, and adjusts the experiment. Here, it is also possible to acquire a background noise (offset) when the channel is disconnected. The background will automatically be subtracted from the recorded values. When the experiment is set, the operator can start the experiment. The application then performs the experiment according to the procedure flowchart described in the previous chapter and immediately displays the measured values in graphs and in numerical indicators. The measured data are written to the output file and supplemented with formatted details about the experiment for further processing by external applications. At the end of the experiment, the CL emission data are automatically corrected for distortion caused by the equipment limitations.

V. MEASURED DATA CORRECTIONS

The measured CL emission intensity can be distorted due to the technical limitations of the experimental equipment. Fortunately, the distorted data can be corrected to some extent using the instrument correction functions. These functions can be determined experimentally, or theoretically in some cases. The software for CL experiments can use the correction functions and automatically correct the measured values of the CL emission, saving the operator time and work.

Performing the efficiency or spectral measurements, it is appropriate to correct fluctuations in the CL emission arising from the fluctuation of the excitation e-beam current. These undesirable short-period fluctuations of the current are typical for termo-emission cathodes. Supposing the fluctuations are relatively small, the increasing current increases the intensity of the CL emission linearly. Therefore, the CL emission intensity can be corrected using the current recorded during the experiment. An example of the difference between the measured and corrected CL emission intensities is shown in Fig. 4. The dashed curve shows the measured CL spectrum of the $\text{Y}_3\text{Al}_5\text{O}_{12}:\text{Ce}^{3+}$ (YAG:Ce) single crystal scintillator. The spectrum was obtained by scanning the visible spectral range using 3 nm wavelength steps. The excitation current fluctuations were in the order of percent during the spectrum scanning. The value of the CL emission intensity and the excitation current was recorded at each scanning step. Using the mentioned values, the mathematical correction of the measured CL spectrum (solid curve) was obtained.

It is usually very important to correct the measured CL spectra for the spectral response of the equipment. The spectral response has its origin in spectral characteristics of relevant parts, such as the spectral sensitivity of a light detector, the spectral efficiency of a spectrometer diffraction grating, and/or the spectral transmittance of a light guide. The spectral characteristics of the individual parts can be determined either from the datasheets supplied by the manufacturer, or, more precisely, by using a calibration lamp with a known emission spectrum. The characteristics can be expressed using a single correction function of the equipment, and the measured CL spectra can be corrected with this function. An example of a correction function application is shown in Fig. 5. The CL spectrum of the YAG:Ce single crystal scintillator (dashed

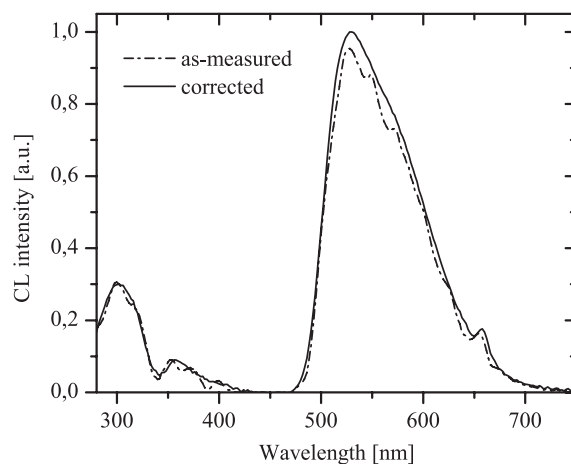


FIG. 4. As-measured CL emission spectrum of the YAG:Ce single crystal (dashed line) compared to the spectrum corrected for the excitation current fluctuations (solid line).

curve) was corrected for the spectral response (solid curve) using the CL equipment correction function (dotted curve).

The examined CL emission time response can be distorted by the time response of the equipment, both after starting and stopping excitation, respectively. The equipment response is caused by the deficient speed of the excitation e-beam modulation, by the detector rise and/or fall time and by the amplifier electronic response. If the responses of the used instruments are not fast enough, they can distort the measured values of the pulsed CL emission. Fortunately, a deconvolution can be used for a correction of the time-dependent CL emission, if the time response of the equipment is known.²⁶ The deconvolution correction function can be obtained either from the documents supplied by instrument manufacturers, or experimentally by using a fast pulsed light source having known characteristics. The as-measured response pulse of the CL emission of the studied YAG:Ce single crystal is plotted in Fig. 6 (dashed curve). The specimen was excited using the 1 kHz excitation pulse with the width of 500 ns. The CL emission signal was deconvoluted using the equipment correction

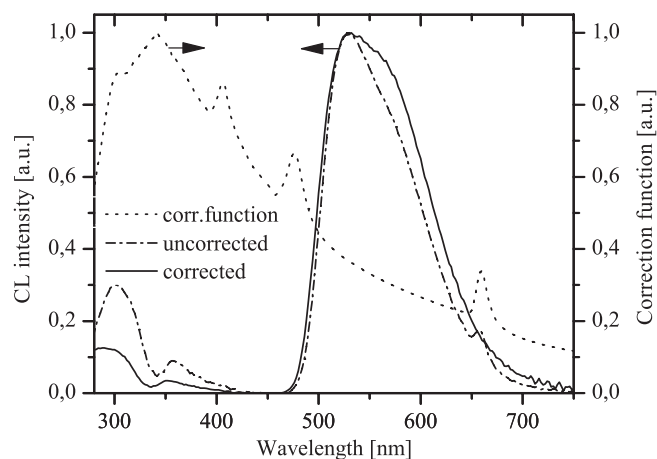


FIG. 5. The CL emission spectrum of the YAG:Ce single crystal uncorrected (dashed line) for the spectral response of the CL equipment (dotted line) compared to the corrected one (solid line).

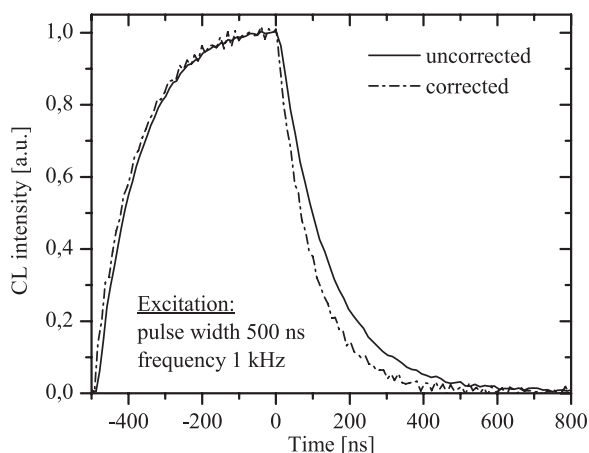


FIG. 6. The CL emission response curve of the YAG:Ce single crystal excited by rectangular electron pulse compared to the curve corrected for the time response of the CL equipment.

function. After the correction, the measured curve changed slightly (solid curve) and its decay time decreased. This confirms the basic idea that the measured decay of the CL emission is slowed down due to the equipment time response.

VI. CONCLUSION

In our laboratory, the cathodoluminescence study of solid materials is performed by using the specialized CL equipment which is characterized by high variability of CL studies, such as CL efficiency, CL spectra, and CL time response studies. For effective performing of all CL experiments, the computer-aided automation was developed and tested. It brought benefits to the experiments and especially to the CL spectral measurements because the manual configuration and control of the spectrometer and the other instruments during the spectra measurement would be considerably difficult. The developed application software for the equipment control and for the measured data acquisition and processing handles many tasks that were not previously feasible in our laboratory. That are, for example, fast and reliable instrument control, well-arranged measured data monitoring, automated experiment initialization and execution, operator error avoidance, multiple data acquisition, and corrections.

The approach of the application software development within the LabVIEW graphical programming platform proved to be very useful. The great LabVIEW features, such as easy readability of source code or utilization of included drivers and libraries, can be motivation for the LabVIEW implementation for the control of CL or similar experiments. A LabVIEW application for instrument control can be developed relatively quickly, if the drivers of the individual instruments are integrated in the LabVIEW, or if the drivers are available by instrument manufacturers. If they are not available (which was a case for the CCD camera used), the development of such control software can be considerably complicated and it could require a lot of effort. However, manufacturer support

of the LabVIEW drivers still grows and it is possible to incorporate more and more instruments to LabVIEW applications.

The presented procedures can be used not only for CL experiments, but also in similar experimental fields, such as fluorescence, electroluminescence, radioluminescence, and other luminescence measurements. During these measurements, an experimentalist should be aware of the technical limitations of an experimental configuration used. These limitations can significantly distort the measured data. If possible, a suitable data correction should be used for accurate data interpretation, such as correction of e-beam current fluctuation, of equipment spectral sensitivity, and of equipment time response. These corrections could be applied in other scientific fields, like optical spectroscopy or time-resolved luminescence.

ACKNOWLEDGMENTS

This work was supported by Grant No. P102/10/1410 from the Czech Science Foundation.

- ¹B. G. Yacobi and D. B. Holt, *Cathodoluminescence Microscopy of Inorganic Solids* (Plenum, New York, 1990).
- ²L. Ozawa, *Application of Cathodoluminescence to Display Devices* (Kodansha, Tokyo, 1994).
- ³P. Schauer and R. Autrata, *Fine Mech. Opt.* **42**, 332 (1997).
- ⁴H. P. D. Schenk, S. I. Borenstain, A. Berezin, A. Schön, E. Cheifetz, A. Dadgar, and A. Krost, *J. Cryst. Growth* **311**, 3984 (2009).
- ⁵S. Boggs and D. Kinsley, *Application of Cathodoluminescence Imaging to the Study of Sedimentary Rocks* (Cambridge University, New York, 2006).
- ⁶R. Autrata, P. Schauer, J. Kvapil, and J. Kvapil, *Scan. Electron Microsc.* **1983**, 489 (1983).
- ⁷P. Horák and P. Schauer, *Nucl. Instrum. Methods Phys. Res. B* **252**, 303 (2006).
- ⁸P. Schauer, *Nucl. Instrum. Methods Phys. Res. B* **269**, 2572 (2011).
- ⁹J. Götze, *Anal. Bioanal. Chem.* **374**, 703 (2002).
- ¹⁰G. F. J. Garlick, *Br. J. Appl. Phys.* **13**, 541 (1962).
- ¹¹F. Bertram, J. Christen, A. Dadgar, and A. Krost, *Appl. Phys. Lett.* **90**, 041917 (2007).
- ¹²M. Merano, S. Sonderegger, A. Crottini, S. Collin, P. Renucci, E. Pelucchi, A. Malko, M. H. Baier, E. Kapon, B. Deveaud, and J.-D. Ganière, *Nature (London)* **438**, 479 (2005).
- ¹³X. Zhang, D. H. Rich, J. T. Kobayashi, N. P. Kobayashi, and P. D. Dapkus, *Appl. Phys. Lett.* **73**, 1430 (1998).
- ¹⁴C. Elliott, V. Vijayakumar, W. Zink, and R. Hansen, *J. Assoc. Lab. Autom.* **12**, 17 (2007).
- ¹⁵I. W. Kirkman and P. A. Buksh, *Rev. Sci. Instrum.* **63**, 869 (1992).
- ¹⁶W. J. Strykowski, *Rev. Sci. Instrum.* **62**, 1921 (1991).
- ¹⁷D. G. Sporea and R. A. Sporea, *Rev. Sci. Instrum.* **76**, 113110 (2005).
- ¹⁸R. A. Aspey, I. S. McDermid, T. Leblanc, J. W. Howe, and T. D. Walsh, *Rev. Sci. Instrum.* **79**, 094502 (2008).
- ¹⁹C. G. Wiegstein, K. H. Schulz, and J. Scott, *Rev. Sci. Instrum.* **69**, 3707 (1998).
- ²⁰L. Kirkup, W. Kalceff, and G. McCredie, *Rev. Sci. Instrum.* **77**, 046107 (2006).
- ²¹T. K. Starn, N. N. Sesi, J. A. Horner, and G. M. Hieftje, *Spectrochim. Acta, Part B* **50**, 1147 (1995).
- ²²J. Ballesteros, J. I. Fernández Palop, M. A. Hernández, R. Morales Crespo, and S. Borrego del Pino, *Rev. Sci. Instrum.* **75**, 90 (2004).
- ²³S. H. Moon and M. D. Foster, *Scanning* **27**, 44 (2005).
- ²⁴D. H. Morse, A. J. Antolak, G. S. Bench, and M. L. Roberts, *Nucl. Instrum. Methods Phys. Res. B* **158**, 146 (1999).
- ²⁵R. Jamal, *Nucl. Instrum. Methods Phys. Res. A* **352**, 438 (1994).
- ²⁶A. E. McKinnon, A. G. Szabo, and D. R. Miller, *J. Phys. Chem.* **81**, 1564 (1977).