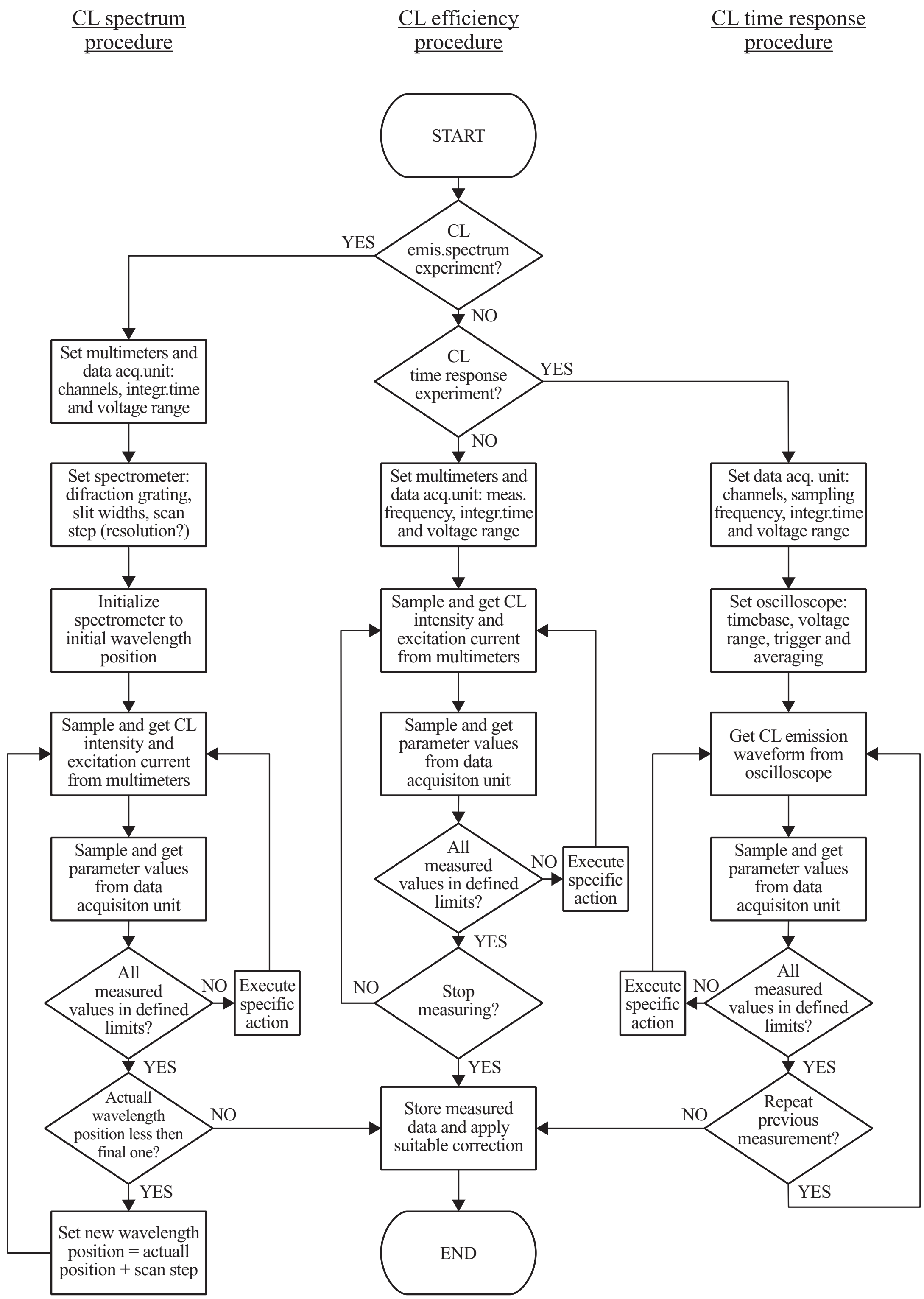

FIG. 2. Procedure flowchart of the CL experiments.

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 application. 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

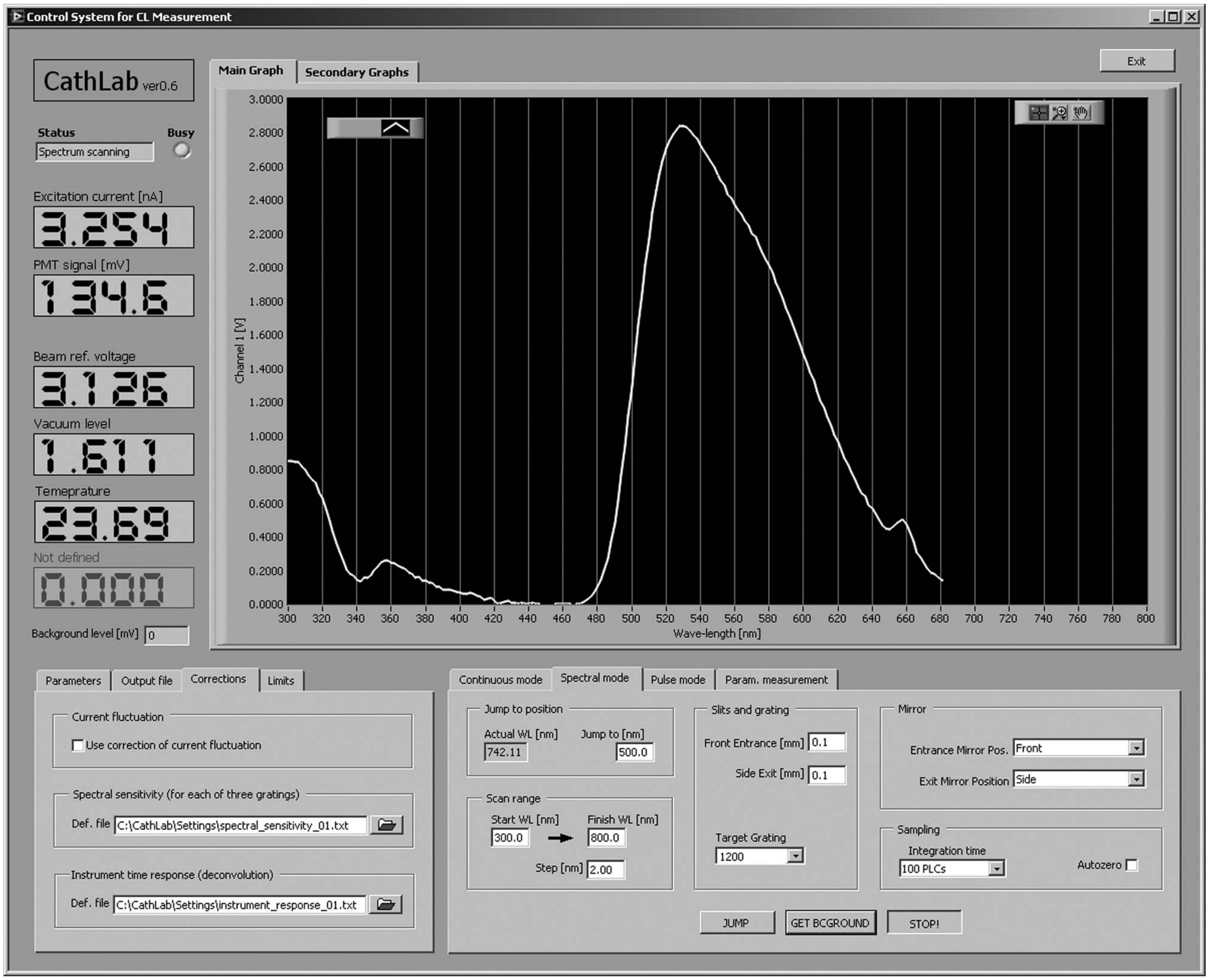

FIG. 3. The application software MS Windows GUI for the CL experiment control and for data acquisition and processing. The actual measurement of the CL emission spectrum of the YAG:Ce single crystal scintillator is shown.

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 $Y_3Al_5O_{12}:Ce^{3+}$ (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 curve) was corrected for the spectral response (solid curve) using the CL equipment correction function (dotted curve).

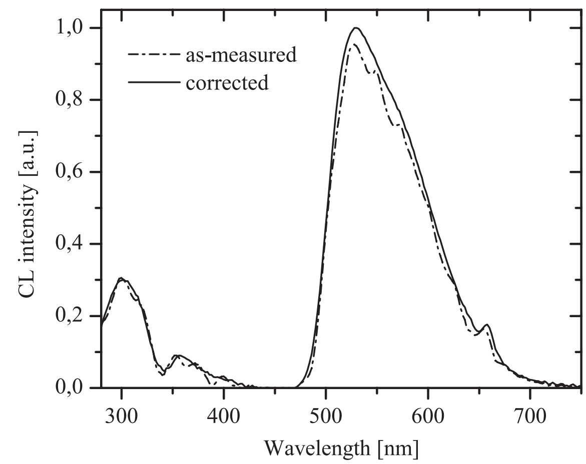

FIG. 4. As-measured CL emission spectrum of the YAG:Ce single crystal (dashed line) compared to the spectrum corrected for the excitation current fluctuations (solid line).

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

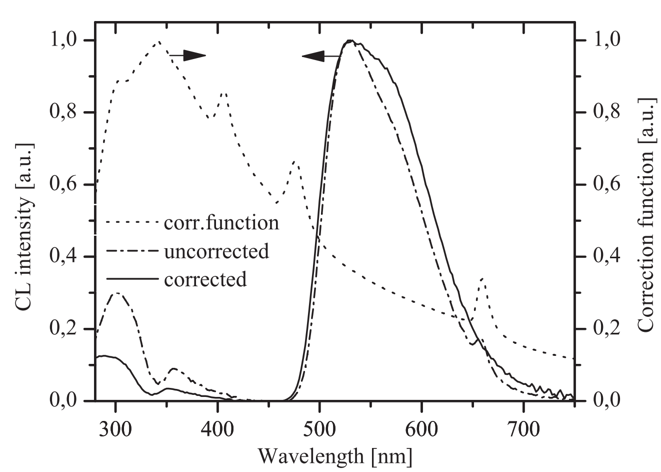

FIG. 5. The CL emission spectrum of the YAG:Ce single crystal uncorrected (dashed line) for the spectral response of the CL equipment (dotted line) compared to the corrected one (solid line).

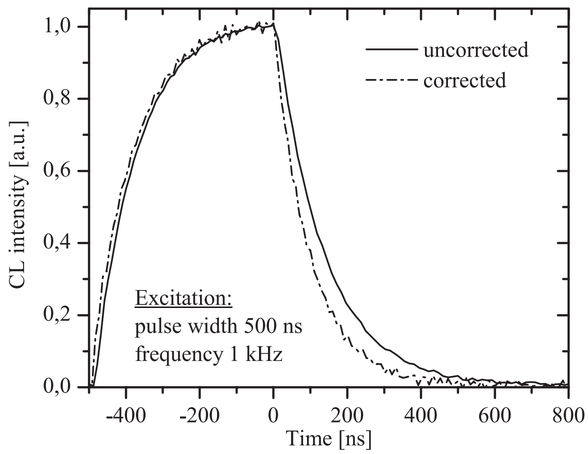

FIG. 6. The CL emission response curve of the YAG:Ce single crystal excited by rectangular electron pulse compared to the curve corrected for the time response of the CL equipment.

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.

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