Recent advances in time-resolved luminescence spectroscopy at MAX IV and PETRA III storage rings

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Recent advances in time-resolved luminescence spectroscopy at MAX IV and PETRA III storage rings

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Abstract. Short-wavelength synchrotron radiation excitation has been an indispensable tool in the studies of the properties of wide gap materials using time-resolved low-temperature luminescence spectroscopy. In recent years, several setups for such investigations have been launched at MAX IV Laboratory and Photon Science at DESY. Two permanently stationed time-resolved luminescence setups at FinEstBeAMS and P66 beamlines are in operation at MAX IV 1.5 GeV and Petra III storage rings, respectively. Mobile luminescence setups have been developed for studies at FemtoMAX and P23 beamlines. FinEstBeAMS, P66 and P23 provide time resolution from ~160 to 100 ps. The FemtoMAX photon source based on an in-vacuum undulator getting an electron beam from the 3 GeV linear accelerator provides an exceptional time resolution of ~30 ps, limited by time response of the photodetector. The performance of the setups, achieved milestones and research challenges are discussed for four new luminescence stations available for the research community with the main focus on time-resolved techniques.

1. Introduction

Short-wavelength synchrotron radiation excitation has been an indispensable tool in studies of wide gap materials using time-resolved low-temperature luminescence spectroscopy [1]. Research activities at the SUPERLUMI station, which was operated in the vacuum ultraviolet (VUV) spectral range at the DORIS storage ring of HASYLAB, DESY during 1985 - 2012, resulted in over 1100 papers published during its 30 years' glorious history and are still published in journals up to the current year. This indicates a tremendous demand for such kind of research facilities contributing to the development of optical and luminescent materials for very different applications. Looking into the field of scintillator research, it is obvious that the studies of materials relevant to nuclear security, fast timing, fast X-ray and medical imaging, neutron detection, high energy calorimetry and scintillation pulse detection need advanced characterisation methods with high time resolution in a wide energy range [2].

Alongside with the dedicated SUPERLUMI station [3], there were several mobile luminescence setups in operation at MAX-Lab (BL-52 at MAX I [4], I3 FinEst branch line at MAX-III [5]), but none of them had such capacity and complexity as the SUPERLUMI luminescence setup at Strahl I.

New 4th generation MAX IV storage rings (1.5 and 3 GeV) in Lund, Sweden and renewed Petra III storage ring (6 GeV) in Hamburg, Germany motivated luminescence community to seek new experimental opportunities at various up-to-date beamlines covering energy range from UV-VUV to hard X-rays. The present paper focuses on the recent developments of dedicated setups for time-resolved luminescence spectroscopy at the FinEstBeAMS [6] and P66 [7] beamlines and mobile setups designed for the FemtoMAX [8] and P23 [9] beamlines at MAX IV Lab and Petra III, respectively. The achieved performance and related experimental challenges will be discussed.

2. Luminescence setup at the FinEstBeAMS beamline at MAX IV Lab

The Estonian - Finnish grazing incidence beamline FinEstBeAMS at the 1.5 GeV storage ring of MAX IV Lab provides a high photon flux of VUV-XUV radiation for various experiments in the energy range of 4.5 – 1300 eV from an elliptically polarizing undulator source. Applying horizontal polarization, the beamline delivers above $2*10^{11}$ photons/s in the photon energy range below 10 eV with a resolving power of 3000, and above 10^{13} photons/s at a resolving power of 5000 in the photon energy range 50–150 eV [10]. This is significantly above the limits of the best performing beamlines based on bending magnets like SUPERLUMI, where the photon flux was typically up to 10^{10} photons/s in the 4-40 eV range, depending on the diffraction grating conditions during 1997-2012 [11]. The grazing incidence optical scheme makes a wide energy range available for the FinEstBeAMS beamline, which is equipped with an end-station for low-temperature (T=5-350 K) photoluminescence research in the UV-visible spectral region (see details in [12]). Such geometry does not suppress higher order radiation, which sets luminescence spectroscopy in a vulnerable position, because the luminescence recorded under the first order excitation cannot be distinguished from that excited by higher orders, as it has already been pointed out in [13]. A set of filters has to be used (optical windows of fused silica (FS) and MgF_2, as well as thin In, Sn, Mg, Al metal foils) in order to suppress higher order radiation. It significantly complicates data processing as one has to compose excitation spectra from segments recorded through different filters taking into account regarding significantly varying transmission of the incident beam from tens to a few percent. Incident photon flux for normalization of the excitation spectra is recorded with an AXUV-100G diode mounted in the experimental chamber.

In addition, a white soft X-ray background is always present, as it is unavoidable for an undulator source in combination with the grazing incidence primary monochromator. Therefore, some residual luminescence is always detected below the respective excitation thresholds in comparison with the beamlines operating with normal incidence monochromators, for which the cut off energy is 40 eV due to the reflectivity of coatings. This is confirmed by our results shown in Fig. 1, where weak but detectable cross-luminescence (CL) is revealed in the UV region below 250 nm under the 17 eV excitation, which is far below the CL threshold at 18.2 eV reported for BaF_2 [14]. It results from the white soft X-ray background and cannot be suppressed by any metal filter available at FinEstBeAMS.

Luminescence from samples in vacuum is collected by an adjustable optical fibre (~0.3 m), which transmits signal through optical feedthrough to the second fibre (~1.5 m) attached to the spectrometer. There is no focusing optics used because the focal spot on the sample 100×100 μm^2 matches well with the fibre size. It is a convenient way to outcouple luminescence, but one has to account for increased absorption in the fibre below 250 nm. In recording decays and time-resolved emission spectra, the fibres can introduce some reflection of the signal disturbing measurements in the ns time range, which can be avoided if luminescence is detected through a set of bandpass or interference filters with an ultrafast photomultiplier tube (PMT) mounted on the FS window of the experimental chamber.

Luminescence can be analysed in the UV-visible-NIR spectral range (200 – 1500 nm) using an Andor Shamrock (SR-303i) 0.3 m spectrometer equipped with three 300 l/mm gratings blazed at 300, 500 and 1200 nm. A variety of exchangeable photomultipliers is used in one spectrometer port with an exit slit, covering spectral range of emissions from 200 to 1350 nm, and a Newton DU9 70P-BVF CCD detector is mounted in the imaging port.

2.1. Results

Fig. 1 shows time integrated emission spectra of a BaF_2 crystal excited below and above the Ba 5p core level ionization energy (at 18.2 eV [14]) by the 17 and 50 eV photons, respectively. The 220 nm emission is due to cross-luminescence resulting from radiative recombination of valence electrons with the Ba 5p core holes [15]. The band peaked at 300 nm is due to triplet self-trapped exciton (STE) emission with a lifetime of 630 ns at room temperature (RT 296 K), whereas that for CL is 0.9 ns (see [16] and references therein).

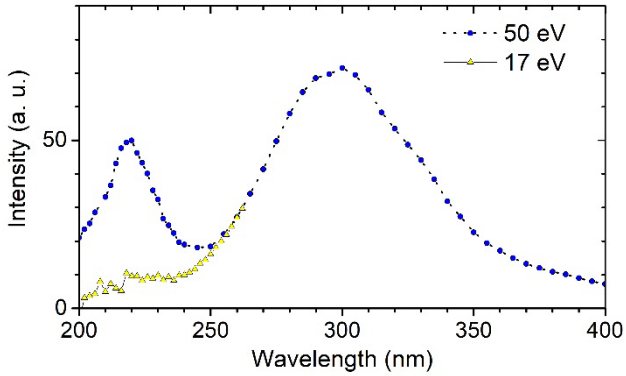

Figure 1. Emission spectrum of a BaF_2 crystal at 7 K, recorded under excitation by 50 eV (blue circles) and 17 eV (yellow triangles) photons at the FinEstBeAMS beamline. The emission spectra were corrected to the transmission of detection channel and sensitivity of photodetector. Mg and Sn filters were applied to remove higher order radiation at these excitation energies, respectively.

In 2020, a sub-nanosecond time resolution option has been implemented by us with a possibility to record luminescence decay kinetics in a single bunch operation mode [17]. Automated acquisition of decay kinetics data during monochromator scanning provides time-resolved emission and excitation spectra in a "spectral decay mapping" mode, which is important for the investigation of excited state dynamics in scintillators. In a single bunch operation mode, the instrumental response function (IRF) as short as 160 ps has been achieved [17].

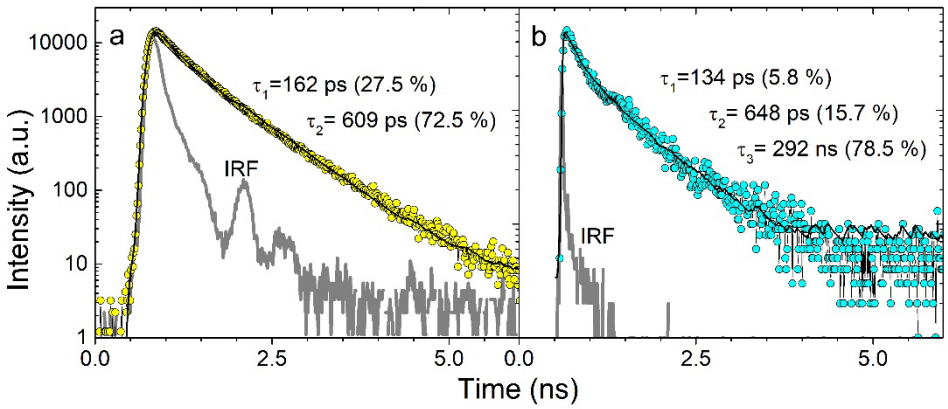

Figure 2. Decay curves of cross-luminescence for a BaF_2 crystal recorded through a 216 nm interference filter (IF216) at 7 K under excitation by 18-23 eV photon pulses (~ 160 ps) at the FinEstBeAMS (with the Sn filter applied) (a) and at 296 K excited by 10 keV pulses shorter than 200 fs at FemtoMAX (b). The decay curves were fitted with exponential decay functions (solid black lines) by deconvolution with a corresponding IRF and the obtained decay times together with their relative contributions are shown in the graphs. Very different IRFs with FWHM values of 160 ps and 28 ps are shown for both setups with a solid grey line. The same ultrafast MCP-PMT (Hamamatsu R3809U-50) was used as a photodetector in both experiments.

Figure 2 shows decay curves of CL from a BaF_2 crystal recorded through an IF216 filter excited by VUV photon pulses (~ 160 ps) at FinEstBeAMS (panel a) and 10 keV photon pulses at FemtoMAX (panel b). Under the VUV excitation, two decay components were identified at 7 K. The main component with τ_2=609 ps constituting near 73% of the total CL intensity is shorter than 920 ps reported in [18] under analogous conditions. The value of the decay time τ_1=162 ps of the faster component coincides with the IRF value of 160 ps and therefore is not correct. As will be shown below, the real τ_1 value could be estimated only in the experiment at the FemtoMAX beamline characterized by better time resolution.

3. Mobile luminescence setup at the FemtoMAX beamline at MAX IV

FemtoMAX is a beamline located at the Short-pulse facility (SPF) at the MAX IV laboratory (Lund, Sweden) and driven directly by the 3 GeV LINAC [8]. FemtoMAX currently generates hard X-ray photon pulses shorter than 200 fs with energy from 1.8 to 12 keV at 10 Hz repetition rate. This makes it well suited for the studies of ultrafast luminescence processes under the excitation simulating real conditions of scintillator operation with an exceptional 28 ps FWHM of IRF limited by time response of the PMT [19].

A special setup was designed for studies at FemtoMAX, with the main aim to achieve efficient luminescence detection of low intensity emissions without compromising time resolution. The 10 keV X- ray photons arrive at the sample through a 4 mm hole in the off-axis parabolic mirror (focal distance FD 25.4 mm). The sample is mounted onto a cold finger of a Janis VPF-800 LN cryostat (T=77-800 K) operating in high vacuum conditions. The same mirror, mounted in front of the sample holder, collects luminescence and directs it as a parallel beam out of the vacuum chamber through a FS window perpendicular to the incident X-rays. With the help of a plane mirror and a second off-axis parabolic mirror (FD 76.2 mm), mounted on the optical table, luminescence is focused to the entrance slit of an Andor Shamrock SR-303i spectrometer (from FinEstBeAMS) equipped with Hamamatsu R3809U-50 MCP-PMT (the same PMT which is used in single bunch experiments at FinEstBeAMS and P23) in one port and a CCD camera in the imaging port. Signals from the MCP-PMT are amplified by a SHF 100 APP preamplifier (12 GHz, 19 dB) and digitized by a LeCroy LabMaster 10-36Zi oscilloscope (36 GHz, 80 Gs/s) belonging to the FemtoMAX equipment. The signal is then further processed by the advanced multi photon counting technique, which allows the detection of several non-overlapping luminescence photons per single excitation pulse. A special LabView software was developed at Tartu University in order to perform this task.

Depending on the lifetime of studied emissions (in the sub-nanosecond time range) a typical count rate is kept far below one photon detected per X-ray exciting photon, making the studies very time consuming. The luminescence spectra can be studied using the CCD detector, but only for bright scintillators like $Y_3Al_5O_{12}$:Ce. Therefore, the spectroscopic research of the samples studied has to be carried out in other beamlines (*e.g.*, FinEstBeAMS, P66 for VUV and P23 for X-ray excitation) located at storage rings, where the number of exciting photons and repetition rates are considerably higher.

3.1. Results

The study of the CL decay kinetics of BaF_2 crystal at 296 K using FemtoMAX by 10 keV photon pulses revealed that the fastest component has the decay time τ_1=134 ps. It is followed by a component with τ_2=648 ps (Fig. 2b), which is comparable with that under the VUV excitation (Fig. 2a). The IF216 filter transmission extends up to 300 nm, covering a short wavelength part of the STE emission (see Fig. 1). Therefore, a third component with the decay time τ_3=292 ns is also detected. This result demonstrates that the time resolution provided by storage rings is not sufficient for studying CL decay kinetics in the sub-nanosecond range, but it is good at the LINAC based sources like FemtoMAX at MAX IV Lab and FLASH at DESY, where the properties of BaF_2 scintillator under high density excitation in XUV were studied [20].

Figure 3 shows decay curves of cross-luminescence from a BaF_2:La 1% crystal recorded at 295 and 80 K for the 225 nm emission through the spectrometer. This way, only CL is detected without any disturbing influence of the STE emission. It is well known that CL intensity and decay times have no temperature dependence [15], which is confirmed also by our experiments. A two-exponential fit for decay curves at both temperatures results in an ultrafast component τ_1=114 ps and a slower one with τ_2=827 and 842 ps, respectively. The second component in La doped BaF_2 is slightly longer in comparison with the CL decay (~ 600 ps) in pure BaF_2. Based on this solid experimental result, we can firmly state that there is an ultrafast decay component, which has to be involved into the description of relaxation processes of core excitations in materials with cross-luminescence. This, however, is beyond the scope of the present paper. The CL decay curves can be well approximated with a sum of two exponentials pointing to the fact that no non-linear phenomena due to mutual interaction of dense core excitations are observed. To take into consideration the experimental parameters such as 120×120 μm^2 spot size on a sample, number of photons $1.5*10^6$ ph/pulse and literature data for the absorption of 10 keV photons in BaF_2, the estimated core excitations density is ≤ $2.5*10^{15}$ cm^{-3}, which is low enough to be in a linear excitation regime without the distortion of decay kinetics. In principle, if the same absorbed X-ray photon creates spatially correlated core holes, their mutual interaction may lead to the appearance of ultrafast component (~ 110 ps) in the decay of cross-luminescence, which is significantly shorter than 600-900 ps reported earlier [14, 15, 16, 18]. Although, strong non-exponential decay components have been observed for various scintillators under high density excitation in the experiments at FLASH [20], which is less probable under the excitation conditions at FemtoMAX as discussed above, but creation of two core holes in the same ion is still plausible even at such excitation densities To answer this question additional studies are needed applying ultrashort excitation pulses at various excitation densities, which can be provided by modern free electron lasers.

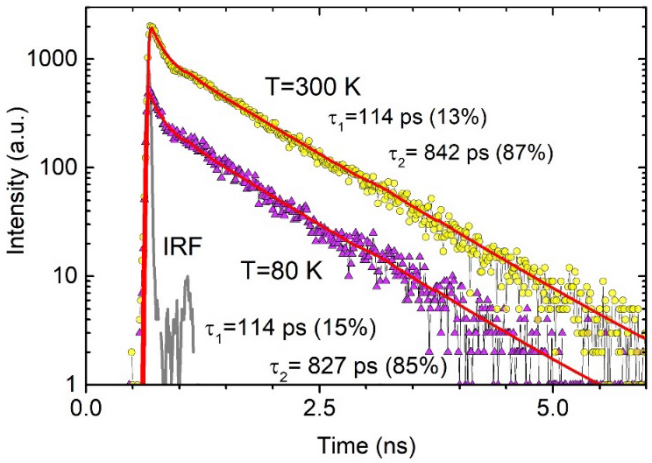

Figure 3. Decay curves of cross-luminescence at 225 nm (spectrally selected by a monochromator) for a BaF_2: La 1% crystal at 295 K (yellow circles) and 80 K (purple triangles) excited by 10 keV photon pulses shorter than 200 fs at the FemtoMAX beamline. The decay curves were fitted with a two-component exponential decay function (red lines) by deconvolution based on the IRF with 32 ps FWHM (a grey line). The decay time values are also shown alongside with their relative contributions.

The luminescence setup at the FemtoMAX beamline has provided high quality data for the research of novel scintillation materials based on nanoplatelets as time taggers [19] and for developing an advanced model of relaxation processes in the well-known CeF_3 scintillator [21].

4. Mobile luminescence setup at the P23 beamline at PETRA III

P23 has a spectroscopy type undulator as an X-ray source providing up to 10^{13} photons in the energy range 5 to 50 keV [22, 23]. In its standard equipment, the P23 beamline has also an Andor Shamrock

303i spectrometer, which is operating for luminescence spectroscopy in the red-NIR spectral region with three gratings 1200, 600, 300 l/mm gratings blazed at 850, 800 and 860 nm, respectively. It is equipped with a spectroscopy CCD iVAC 316 LDC-DD camera as a detector. The goal of our research was to study the non-proportionality of scintillators as a function of the X-ray excitation energy, monitoring the changes in luminescence decay curves, which are sensitive to local excitation density. For this purpose, a mobile setup for time-resolved luminescence was developed (see Fig. 4a). It consists of a Janis VPF-800 LN vacuum cryostat, an Andor Kymera 328i UV-visible spectrometer (provided by the P66 team), a Hamamatsu R3809U-50 MCP-PMT photodetector, signal processing electronics comprising an ORTEC 9327 discriminator and a Chronologic xTDC4 time-to-digital converter with the developed software, which is integrated into the beamline control system for recording time-resolved excitation spectra. For the measurements of decay curves and time resolved emission spectra, a separate software based on LabView is applied. X-ray excited luminescence is collected through a cryostat window and focused using a lens system into the entrance slit of the spectrometer. The setup provides temperature control in the range of 77-800 K and time-resolved luminescence detection in the spectral range 200-850 nm. The actual time resolution IRF of the whole system depends on the electron bunch length and bunch-to-bunch stability and 105 ps FWHM was achieved in the 40-bunch operation mode of Petra III storage ring.

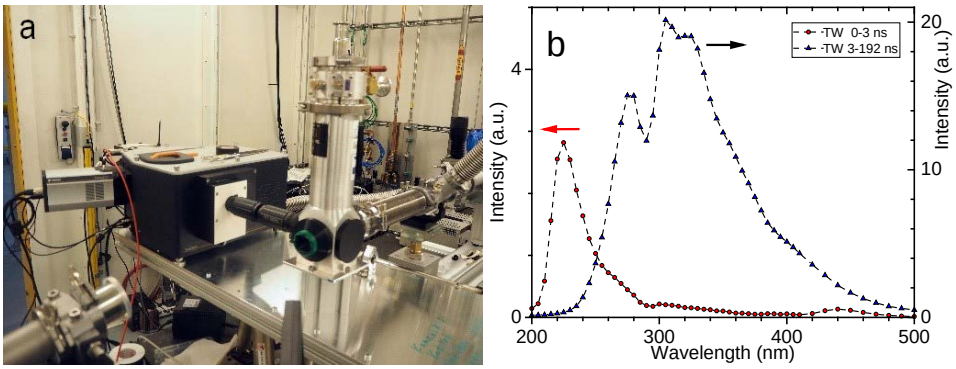

Figure 4. (a) Photo of the mobile luminescence setup mounted at the P23 beamline at Petra III storage ring, DESY Photon Science. (b) Time-resolved emission spectra of BaF_2 crystal at 295 K in two different time windows of 0-3 ns (red symbols) and 3-192 ns (blue symbols) excited by the 9 keV photons. The emission spectra are presented as recorded.

An example of the performance of this easy to handle setup is shown in Fig. 4b, which depicts time-resolved emission from a BaF_2 crystal at 295 K. To obtain the emission spectra, luminescence decay curves were recorded at each emission wavelength. The emission spectra are presented in two different time windows, short (STW with time range 0-3 ns) and long (LTW, 3-192 ns), obtained by integrating signal intensity in the marked intervals. The length of LTW is limited by the interval between successive excitation pulses. In STW, CL at 220 nm is dominant, whereas the STE emission at 300 nm is observed in LTW. The dip near 290 nm is tentatively assigned to radiation induced absorption.

5. Luminescence setup at the P66 beamline at PETRA III

A new P66 beamline at PETRA III storage ring was set into operation in late 2021 (see Fig. 5a). The P66 beamline was relocated and refurbished from the old SUPERLUMI station at DORIS synchrotron [24]. Despite a fact, that the P66 beamline dedicated to luminescence research has been completely modernized, its main features are similar to SUPERLUMI capacity. The photons from a bending magnet are collected by two mirrors and focused on the entrance slit of the normal incidence primary monochromator, with a 2 m focal length unit in 15° McPherson mounting. Its linear dispersion is 4

Å/mm provided by two 1200 l/mm holographic concave gratings coated by Al and Pt, which cover the energy range of 4-40 eV with the suppression of higher orders below 20 and 40 eV, respectively.

Fig. 5b shows the photon flux recorded using sodium salicylate luminescence. Two gratings together with MgF_2 and fused silica filters provide nearly higher order free VUV photon beam for studies. The slits of the primary monochromator (FMB Berlin) are continuously adjustable between 10 µm and 2 mm. The third mirror focuses monochromatic radiation onto a sample holder attached to a flow-type liquid helium cryostat (T=5-350 K). For analyzing luminescence, a Kymera-328i UV-visible spectrometer is used with three interchangeable gratings (300 l/mm), which are blazed at 300, 500 and 1200 nm. Hamamatsu PMTs R6358 PMT (IRF ~700 ps) and R3908U-50-MCP (IRF ~100 ps) can be used for time-resolved spectroscopy and a Newton 920 CCD detector is destined for high resolution luminescence spectroscopy. A more detailed description of the P66 beamline can be found at the DESY confluence web page [7]. Another secondary monochromator for luminescence analysis in the VUV range (4.75 – 12 eV), which is equipped with a solar blind PMT R6836 from Hamamatsu and an ultrafast open microsphere plate detector (IRF < 100 ps), is under commissioning. This is a unique feature of P66 currently not available in any other luminescence station.

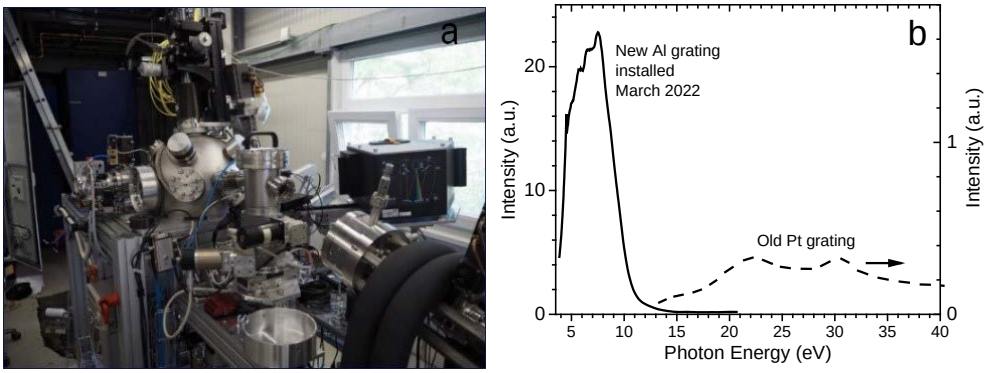

Figure 5. (a) Photo of the new dedicated time-resolved luminescence setup mounted at the P66 beamline at Petra III DESY Photon Science. (b) Relative photon fluxes used for normalization were recorded from sodium salicylate for the newly installed Al coated grating blazed at 180 nm (solid line) and for the Pt coated grating (dashed line) covering higher energy range 20-40 eV, which was in operation at the SUPERLUMI station up to 2012 and is still of good quality for VUV spectroscopy.

6. Conclusion

There are two permanent luminescence stations in operation at synchrotron facilities in Europe: P66 covering 4-40 eV in normal incidence geometry and FinEstBeAMS operating at 4.5-1300 eV in grazing incidence geometry. Two mobile luminescence stations are implemented for studies at X-ray beamlines P23 and FemtoMAX. All luminescence stations possess capacity for time-resolved luminescence spectroscopy and recording decay curves with IRF time resolution from 30 to 160 ps. These developments contribute significantly to the research of novel scintillators and optical materials providing high quality data acquisition.

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References

[1] Zimmerer G 2007 *Rad. Measurements* **42** 859
[2] Dujardin C, Auffray E, Bourret-Courchesne E, *et al.*, 2018 *IEEE Trans. Nucl. Sci.* **65** 1977
[3] Möller T, Gürtler P, Roick E, Zimmerer G 1986 *Nucl. Inst. Meth. Phys. Res. A* **246** 461
[4] Kink R, Löhmus, A, Niedrais, H, *et al.*, 1991 *Phys. Scripta* **43** 517
[5] Feldbach E, Avarmaa T, Denks V P, *et al.*, 2013 *Phys. Scripta* **T157** 01401.
[6] Pärna R, Sankari R, Kukk E, *et al.*, 2017 *Nucl. Inst. Meth. Phys. Res. A* **859** 83
[7] https://confluence.desy.de/display/FSP66/P66+Time-resolved+luminescence+spectroscopy
[8] Enquist H, Jurgilaitis A, Jarnac A, *et al.*, 2018 *J. of Synch. Rad*. **25** 570
[9] https://photon-science.desy.de/facilities/petra_iii/beamlines/p23_in_situ_x_ray_diffraction_and_imaging /index_eng.html
[10] Chernenko K, Kivimäki A, Pärna R, *et al.*,2021 *J. Sync. Rad.* **28** 1620
[11] Zimmerer G, a personal communication
[12] Pankratov V, Pärna R, Kirm M, *et al.*, 2019 *Rad. Measurements* **121** 91
[13] Beaumont J H, Bourdillon A J and Kabler M N, 1976 *J. Phys. C: Solid State Phys.* **9** 296
[14] Kirm M, Lushchik A, Lushchik Ch, Nepomnyashikh A I, Savikhin F, 2001 *Rad. Meas.* **33** 515
[15] Makhov V N 2014 *Phys. Scripta* **89** 04401
[16] Dorenbos P, Visser R, Dool R, *et al.*, 1992 *J. Phys.: Condens. Matter* **4** 5281
[17] Saaring J, Vanetsev A, Chernenko K 2021 *J. Alloys Compd.* (2021) **883** 160916
[18] Terekhin M A, Vasil’ev A N, Kamada M, Nakamura E, Kubota S 1995 *Phys. Rev B* **52** 3117
[19] Turtos R M, Gundacker S, Omelkov S, *et al*., 2019 *npj 2D Materials and Applications* **3** 37
[20] Kirm M, Nagirnyi V, Vielhauer S and Feldbach E 2011 *SPIE Proc.* **8077** U1
[21] Kamenskikh I, Tishchenko E, Kirm M, *et al.*, 2020 *Symmetry* **12** 914
[22] Rönfeldt P, Svensson Grape E , Inge A K, *et al.*, 2020 *Inorg. Chem.* **59** 8995
[23] Ruiz Arana L, Ströh J, Amtsfeld J, *et al.*, 2022 *Zeitschrift für Naturforschung B*
[24] Wilcke H, Böhmer H, Haensel R, Schwentner N 1983 *Nucl. Inst. Meth. Phys. Res. A* **208**, 59