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FOR LUMINESCENCE MEASUREMENT

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ABSTRACT

A new experimental set up for time and spectrally resolved luminescence experiments on atoms, molecules and solids under state selective excitation with synchrotron radiation (SR) is described. A uniquely large spectral range of luminescence analysis from 50 to 1000 nm is covered by a combination of two monochromators. The central device is a specially designed VUV toroidal grating monochromator with an extremely large f-number 1 : 2.8 for a working range from 50 to 300 nm. Excellent agreement between calculated and measured performance of this instrument is found.

"Now at DESY-HASYLAB, 2000 Hamburg, Germany

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1. INTRODUCTION

Luminescence experiments in the VUV under state selective optical excitation suffer very much from intensity problems. SR of a storage ring turned out to be a powerful excitation source for such experiments¹. In the new SR laboratory HASYLAB a high intensity high resolution VUV beamline for luminescence experiments has been constructed².

In this paper, some features of a multipurpose experimental set up for time and spectrally resolved luminescence at the above mentioned beamline are described. The essential properties of the set up are

- (i) spectral analysis of luminescence in an extremely large spectral range from 50 to locorm
- (ii) time resolution of spectrally selected luminescence under state selective excitation down to the 50 ps range
- (iii) sample preparation in an UHV environment including growth of cryocrystals and in situ cleavage of crystals at low temperatures (down to 4 K)
- (IV) interchangeable sample holders for solid and gas phase samples
- (V) simultaneous recording of high resolution absorption and excitation spectra

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2. GENERAL LAYOUT OF THE SET UP

Figure 1a shows a top view of the set up including the monochromator PM of the beamline²⁾. The set up consists of the UHV sample chamber SC, and the UHV monochromator chamberMC, separated by an UHV valve V. The sample chambercontains the last element of the beamline, namely the mirror L3 which focusses the exciting light onto the sample (position of the focus at S).

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Luminescence is analysed at right angle to the direction of exciting beam. The monochromator chamber contains the VUV monochromator which is described in detail below. A plane mirror M can be turned into the light path to deflect the zeroth order of grating G upwards. The focus then is outside the vacuum. Luminescence is analysed between 200 and 1000 nm with a commercial monochromator (VIS-SM).

The VUV monochromator has no entrance slit. The source is the luminescent part of the sample (typically 4 mm x.) mm). In this way, intensity losses due to optical fitting of the instrument to the sample with additional elements are avoided (especially important for $\lambda \lesssim 120$ nm)

3. OPTICAL DESIGN OF THE VUV MONOCHROMATOR

The VUV monochromator in chamber MC is the essential device of the set up. Our aim was the construction of an instrument with optimal iuminosityresolution product compatible with several requirements like

- (i) resolution interval $\delta\lambda$ ξ_*5 nm in a working range 50 300 nm
- (ii) limitation of the image size to $h' \leq 10 \text{ mm}$ (due to typical detector areas) for an object size of $h \leq 4 \text{ mm}$ (length of the luminescent spot)
- (iii) geometrical limitations connected with the requirement that the luminescent part of the sample serves as an entrance slit.
 The optical design is based on the P.S.M. 20-R mounting³⁻⁵⁾. This is an aberration corrected instrument with fixed object (r) and image distance (r). 2 θ is the fixed angle between the two beams and R the main radius of curvature of the grating.

Starting from R = 50 cm, the different requirements are met with 2 θ = 28°, r = 391 mm and r = 652 mm $\pm \Delta r$ (λ). Width W of the grating and length L of the grooves can be as large as ∞ 130 mm if a toroidal grating with non uniforme grooves distribution (n = 1650 k/mm) is used. This corresponds to an extremely large f number of 1 : 2,8 in zeroth order which is unique in combination with the other properties of the instrument.

The design rules of the grating include geometrical and physical optics criteria associated with the phase balancing method³⁻⁶⁾. The asymmetrical configuration⁷⁾ ($r \neq r'$) is derived from the second order focussing condition (which allows, via physical optics criteria, a balance of the pair aberration terms) and from optimisation of the luminosity-resolution product. To satisfy the brightness conservation law for the required h' toroidal blank and phase correction by the hologram are combined to reduce astigmatism to less than .o3 L. The coma broadening is reduced by a proper localisation of the two recording laser point sources for the hologram.

4. MECHANICAL REALISATION

Figure 2 shows the layout in more detail. The monochromator consists of the grating holder GH with grating G, the wavelength drive WD, the exit slit ES and the detector assembly DA. All components work without any lubricant and are bakeable. Restrictions concerning baking exist only for G, the deflecting mirror M, and a charnel plate detector C in DA. Nevertheless, pressures below 10^{-9} mbar are reached.

The grating holder is mounted on an extra plate P inside the chamber to avoid readiustments after pumping down. The grating can be rotated in its holder independently around three main axes for a convenient proper adjustment. To do these rotations precisely for such a heavy erating (5 1.5 kg), the axes are guided by special UBV ball bearings.

The wavelength drive is a true sine drive consisting of a lever arm, a linear motion feedthrough and a shalt outside the vacuum. At the end of the lever arm, a stainless steel ball glides on a precision quartz plate on top of the feedthrough. The length of the lever arm is adjustable. 1µ linear motion of the shaft corresponds to a wavelength interval of .ol nm. The linear motion of the shaft is measured by a linear optical encoder with an accuracy of 1µ. Thus the reproducibility (as well as linearity) of the device is better by an order of magnitude than the resolution.

The exit slit consists of two slit jaws mounted on a Cu-Be ring. By deforming the ring with a precision feedthrough, the slit can be opened symmetrically from 0 to 5 mm. The height of the slit is limited by a fixed mask to 12 mm. The detector assembly containes a channel plate detector C inside the UHV for $\lambda \leq 130$ nm. A small mirror M¹ can be moved into the beam with a linear motion feedthrough. It then deflects the beam out of the UHV (through a LiF window). Sutside, under normal vacuum conditions, several other detectors for lenger wavelengths can be used.

Not shown in figure \mathbb{C} are several masks for suppression of scattered light. Moreover, a special mask can be positioned in front of the grating from outside to reduce N to N = 50 mm. In this way, resolution is increased at the expense of intensity in the overall useful spectral range.

5. PERFORMANCE OF THE MONOCHROMATOR

In table 1, the calculated resolution (FWHM) is compared with experimental values for a given size of the entrance slit and a few wavelengths around 250 nm where the grating is optimised. ${}^{3}\lambda_{p}(1)$ is deduced from physical optic (FWHM of image profile assuming triangular shape), $\delta\lambda_{p}(2)$ from geometrical optic (ray tracing method) both of them associated with the phase balancing method. Very good agreement is obtained between theory and experiment.

Figure 3a shows measured FWHM as a function of defect of focus. In figure 3b, the FWHM (crosses) under optimal focussing conditions are compared with calculated curves. Though they are valid for smaller apertures, we clearly see that - under optimal focussing conditions in the whole working range the requirement $\delta\lambda \leq .5$ nm is fulfilled. Normally, however, a fixed value for r' will be used. Figure 4 shows the measured resolution as a function of wavelength for r' = 652 mm (optimal for λ = 250 nm). The size of the entrance slit here was 4 mm x .15 mm. The exit slit width was chosen as s' << FWHM. The crosses are measured for full aperture. Between ~150 and ~250 nm, $\delta\lambda$ is mainly given by the dimension of the entrance slit. Only below and above this range does the aberration in connection with the defect of focus lead to a larger $\delta\lambda$. Consequently, masking of the grating (circles) increases resolution only at short and at long wavelengths. The full curve was calculated for the condition shown by the crosses. Excellent agreement is found.

The insert in figure 4 shows a typical line profile (λ = 115 nm) for full aperture (slightly asymmetric due to coma terms) and reduced aperture (reduction of FWHM and more symmetric line shape).

6. CONCLUDING REMARKS

A VUV monochromator with a very large f-number has been designed for luminescence experiments in connection with SR excitation. The theoretical design parameters are reached. The set up has been successfully used for various luminescence experiments on matrix isolated molecules⁸, on pure rare gas solids and alkali halides (free exciton luminescence⁹) and e.g., on the Cl₂ molecule $(1^{1}\Sigma_{u}^{+} \rightarrow X^{1}\Sigma_{g}^{+})$ bound-bound and bound-free fluorescence)¹⁰. Without the generous financial support of the BMFT this project would not have been realised. The technical staffs of the II. Institute and of HASYLAB helped very much. We especially thank the chief designer of the II. Institute, D. Giesenberg, Miss E. Soltau who made most of the drawings, and the excellent mechanical workshop of the II. Institute, guided by J. Weber. Finally, we especially acknowledge the continous co-operation of our colleagues from the University of Kiel who built up the beamline.

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FIGURE CAPTIONS

- Figure 1 Schematic layout of the set up. Part a: top view, part b: side view along AB. VUV-SM: secondary monochromator for the VUV range. VIS-SM: secondary monochromator for the visible range.
- Figure 2 Detailed layout of the VUV monochromator.
- Figure 3 a) Resolution (FWHM) as a function of defect of focus for
 a few selected wavelengths (W = L = 130 mm)
 b) Resolution (FWHM) as a function of wavelength for different
 apertures and minimal defect of focus.

Figure 4 Resolution (FWHM) as a function of wavelength, for fixed image distance r' = 652 mm, entrance slit 4 mm x .15 mm. A (x): full aperture. The full curve is calculated. B (o): reduced aperture (L = 130 mm, W = 50 mm). C (+): for comparison, a few expt. points are included for minimal defect of focus and entrance slit 4 mm x .06 mm. Insert: line profils of λ = 115 nm for different apertures.

TABLE 1

calculated and experimental FWHM for W = L = 130 mm, entrance slit 4 mm x .06 mm. s' means the exit slit width.

λ (nm)	δλ _. (1) (nm)	^{የኢ} (2) (nm)	$\delta \lambda_{p}^{expt}$.	s * (mm)
296.8	.38	.44	.40	.2
253.6	.17	.30	.24	, 2
237.8	.18	, 31	.27	. 2





Figure 1



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