SOPHISTICATED SYSTEM FOR DETECTION OF LOW-INTENSITY PHOTOLUMINESCENCE FROM NANO-DIMENSIONAL MATERIALS WITH UV EXCITATION

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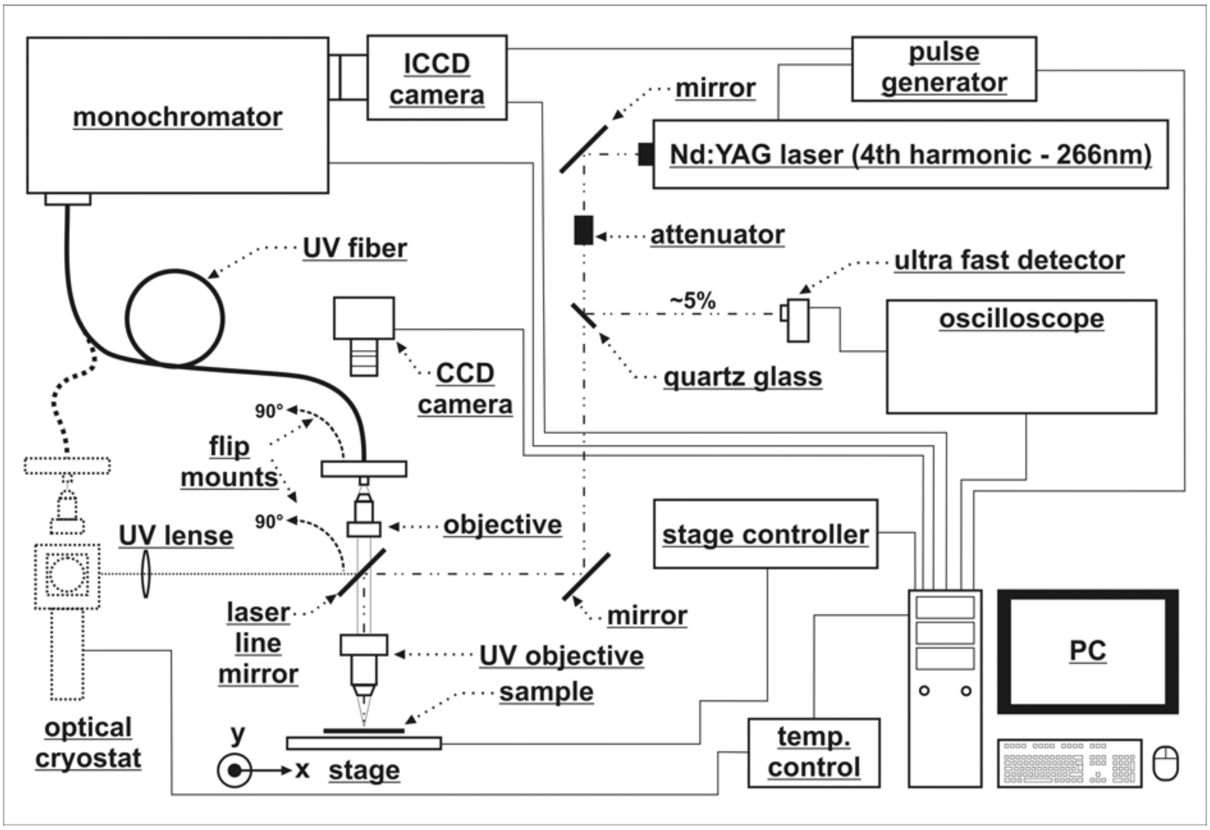
1. Introduction

Recently the material science is proceeding towards low-dimensional particles as a result of their unique properties which cannot be observed in bulk materials. Nanoparticles and nanostructures are nowadays far below diffraction limit of optical visualization devices. Although there are advanced techniques for visualization available such as Scanning Electron Microscopy or Atomic Force Microscopy, there are still characterization techniques for getting an important information on materials which cannot avoid usage of light for analysis. Photoluminescence is such a technique where light is used for excitation to get a response from materials. There are few solutions how to avoid the diffraction limit of light such as Near-field Scanning Optical Microscopy which uses an optical fiber for excitation and/or collection of light. This allows to characterize optical properties of nanostructures with high resolution [1,2], but even though is this method slow and not suitable for characterization of larger areas of nanoribon, nanowire or nanotube overlapping clumps which may act as random nanolasers [3]. For such advanced measurements a system capable of changing laser intensity, moving the sample in the terms of nanometers with highly sensitive detection device and time-resolved mode is needed.

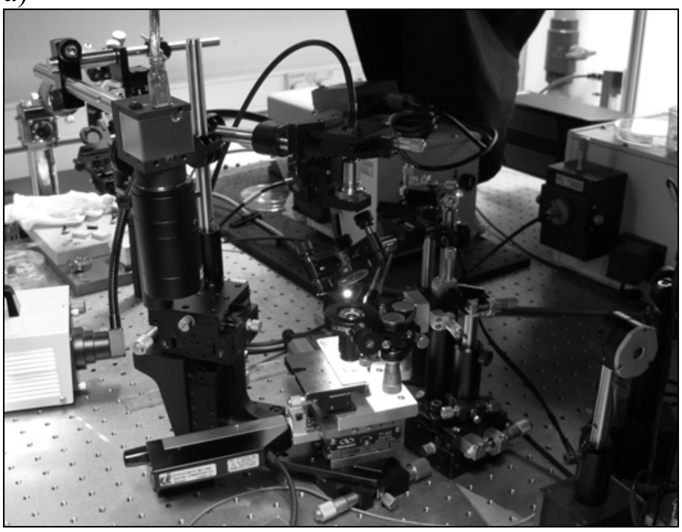
2. Experimental

We have built a sophisticated system for low-intensity PL measurements with short-pulse UV excitation laser (Fig.1a)). The excitation part includes Nd:YAG laser on 4th harmonic frequency (266nm), attenuator M-935-3-OPT for possibility to change laser beam energy, quartz glass for pulse monitoring with ultrafast UV enhanced silicon detector UPD-200-UD connected to Infiniium 54835A dual channel oscilloscope, laser line mirror tuned to high reflection at 266nm for 45° incidence angle and LMU-15x-UVB microscope objective for focusing of laser beam and collection of photoluminescence. The measurement part consists of above mentioned UV microscope objective for irradiation collection, 20x microscope objective for focusing of collected radiation into optical fiber, monochromator ACTON SpectraPro-500i with 150, 600 and 1200gr/mm gratings and ICCD camera PI-MAX from Princeton Instruments with Unigen II intensifier. Moreover, the system consists of devices for measurement and control which are connected and controlled by computer using program created in LabView® environment.

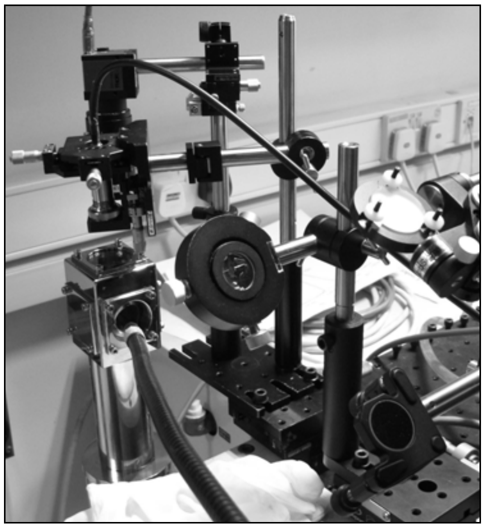
The laser beam is focused onto sample using long working distance UV enhanced objective with the smallest spot size reaching ~10μm which is sufficient for excitation of few micrometer long nanowires, nanoribbons, nanodiscs or clusters of nanoparticles. The same objective is used for light collection which propagates through laser line mirror to 20x microscope objective focusing the light into optical fiber cluster with 1mm diameter (Fig.1b)). Then the light is transported by the fiber into high resolution monochromator and intensified inside fast ICCD camera (in range of 150-900nm) with frame rate up to 5MHz. Width of the laser pulse is monitored by fast oscilloscope (1GHz bandwidth) through ultrafast silicon detector (rise time <175ps) aligned to detect signal from quartz glass



a)



b)



c)

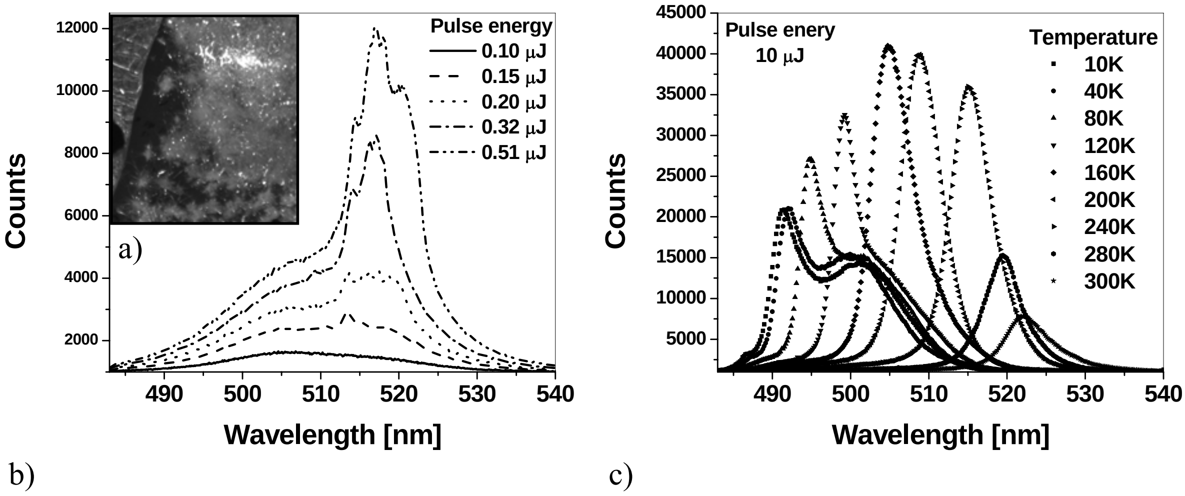
Fig.1: *Sophisticated PL measurement system a) schematic b) room-temperature setup c) low temperature setup.*

reflection (~5% for 266nm) of primary laser beam and constant laser pulse width of ~4ns has been measured. Other purpose of this measurement is to get exact power of the laser in the pulse which is varying for each pulse approximately in a range of 20%. For such measurement an optical power meter was employed to determine calibration constant for integral intensity from ultrafast detector. With this feature the system is able to include exact power information for each pulse in resulting data file. The system is synchronized by delay/pulse generator DG535 for exact timing of laser pumping and camera detection delayed gating, everything controllable by software. For mapping measurement there is ESP300 Motion Controller/Driver available with high load motorized actuators LTA-HL mounted on x-y stage with minimal step of 50nm and the software allows user to configure mesh or line mapping measurement. User may monitor the sample position and incidence spot on the sample by flipping out the fiber holder for optical image from high resolution digital CCD camera DCU223M with 5-50mm focal length adjustable camera objective.

For low temperature measurements is helium closed-cycle optical cryostat available when the laser line mirror is flipped out of the beam propagation path. It is possible to reach 10K inside the cryostat and change the temperature to desired value using PID temperature controller. Moreover, the software in computer interface allows using electrical source/meter for I-V curves during any measurement simultaneously with electroluminescence from light emitting devices such as LEDs or LDs.

Every part of the system is controllable by developed software through computer interfaces. User may monitor the sample by CCD camera and move the sample to required position using the joystick or software. The software also allows user to choose between normal or time resolved measurement where the gating pulse is shifted each measurement in desired time range with gate width up to 10ns and time jog resolution less than 1ns.

As an example of system functionality have been chosen measurements of Cu doped CdS nanoribbons grown on silicon wafer. Sample was synthesized in a horizontal tube furnace with three independently controlled temperature zones with high-purity CdS and Cu_2S powders placed in the middle of the furnace while the silicon wafer substrates pre-coated with 2 nm Au as a catalyst were placed between the last two zones to achieve temperature gradient. Camera image of the sample on Fig.2a) shows dense disordered nanoribbons film with thickness of few millimeters in top right corner and aragonite shaped nanoribbons nests in bottom left corner. The sample has been measured at room temperature under different incidence energies of laser beam (Fig.2b)) and at low temperatures with



b) c)

Fig.2: *CdS:Cu nanoribbons grown on silicon substrate a) camera image of sample b) PL spectra by different incidence energy of laser c) PL spectra by different temperature.*

constant laser beam energy of 10μJ (Fig.2c)). With the nano-positoning system we have been able to find a position at the sample with stable lasing properties. The sample shows narrow photoluminescence peak with resonant modes at 518nm for room temperature measurements related to nano-lasing of CdS nanoribbons visible also in the low-temperature measurements where the peak is shifting up to 490nm as expected. There is no possibility of exact laser spot positioning inside cryostat chamber and thus the spectra obtained at low-temperatures are not showing laser modes. Broad photoluminescence with maximum peak at 500nm is most likely related to defect emission and is permanent at the same wavelength even at low temperatures. These spectra are liable to further research before definite conclusion will be stated.

3. Conclusion

A system capable of low-intensity photoluminescence detection and positioning in terms of tens of nanometers has been built. With UV excitation source and possible attenuation of laser intensity is the system capable of nano-dimensional particles characterization and with a calibrated ultrafast optical detector is the energy of the incidence beam well defined for each measurement. The system is suitable for electrical and optical measurement of light emitting devices such as LEDs, OLEDs or LDs when electric source/meter is employed. By usage of optical cryostat it is possible to perform low-temperature photo or electroluminescent measurements. Furthermore, the system has a great potential for improvements by replacing of some optical parts or by using faster ICCD and oscilloscope for sub-nanosecond time-resolved measurements.

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