Application Note 1084-752

PPMS Fiber Probe: System for photoconductivity measurements in the PPMS

Michael Foos, Robert Janz, Marc Kunzmann, Stefan Riesner, LOT-QuantumDesign GmbH, Germany

Studying sample properties in an illuminated state has become possible in a Quantum Design PPMS by the integration of an optical fiber into a multi-function probe (MFP). This way, a PPMS system can perform automated measurements of samples as a function of temperature, field or illuminated wavelength (or a combination of all). This note introduces a setup consisting of a light-source attached to a motorized monochromator with fiber coupling at the output. A customized fiber assembly fitting to the MFP allows a variable sample illumination while the PPMS analyzes electrical transport properties.

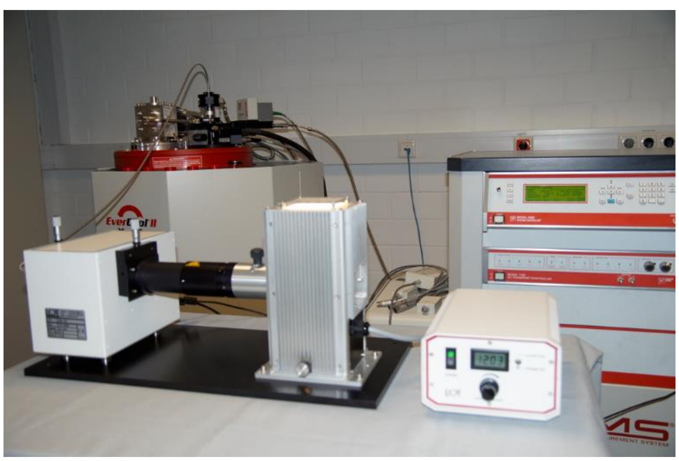

Figure 1: Setup with MFP installed in PPMS-ECII, monochromator (left, front), light source (middle, front), power supply (right, front)

Description of the monochromatic light source

Normally, a laser is considered to be an excellent monochromatic light source. However, lasers are rather expensive and typically provide only a single wavelength. An economic alternative with high flexibility regarding the output wavelength is the combination of a light source and a monochromator. Our choice for the shown setup is a halogen lamp in combination with a 150 mm focal length monochromator. The lamp housing and monochromator are mounted on a single plate with the beam path protected by a tube. At the output, the monochromator has an SMA fiber adapter for easy connection.

The monochromator

Our monochromator model MSH-150 has a Czerny-Turner layout with two alternate gratings. The first grating is used for wavelengths from 350 nm to 1100 nm, and the second grating is for the range from 1100 nm to 2 µm. In addition, an optical filter is placed in the beam path for removing wavelengths from higher diffraction orders. A filter wheel is used to cover the complete wavelength range. The wheel has six positions and is equipped with long-pass filters with 50% cut-offs at 320 nm, 550 nm, 645 nm, 780 nm, 1050 nm, and one closed position called “blank”. The gratings and the filter-wheel are fixed on motorized mounts, which allows computer control of the setup. A corresponding software package is part of the photoconductivity kit.

The light source

The light source consists of a 100 W halogen lamp with a color temperature of ~3200 K. The lamp housing has no reflector to enhance the lamp life-time, which shall be approximately 2.000 hours. The light is collected by a collimator and focused on the entrance slit of the monochromator with a lens made from BK7. The output from the lamp starts around 350 nm. Shorter UV wavelengths are absorbed by the quartz of the lamp. The lamp is powered by a voltage supply which allows manual adjustment of the voltage and thus the light intensity within a certain range.

In LOT’s product portfolio, the combination of a light source with a broad spectral emission range and a monochromator is called monochromatic light source, or short MLS. The MLS provides the user a flexible setup for experiments which use different wavelengths. However, the light is not as purely monochromatic as it would be from a laser. The bandwidth of the output light in the used setup has a width of approximately 10 nm (FWHM).

The fiber and integration to the MFP

We have chosen a quartz fiber, which is optimized for the UV-VIS range. The fiber bore has a 1 mm diameter core and an outer cladding. The fiber has been customized to fit to a standard multi-function probe (MFP). The fiber part which is outside of the PPMS system has a flexible, metallic jacket for mechanical protection. The fiber part inside the PPMS has a length adapted to the length of the MFP. The end of the fiber, which is plane and polished, is a few centimeters above the regular sample location. Since the sample stage in the MFP (model P450A) can be set at different heights, it is possible to change the distance of the fiber output and the sample.

Figure 2 shows the fiber integrated in a multi-function probe model P450A. The fiber in the MFP has no jacket. As a result from the production process, it has a very thin cladding, which increases the mechanical stability of the blank fiber. However, we recognized that thermal cycles, especially at cryogenic temperatures, result in tiny cracks of the cladding but not of the fiber core. Because of this, we have removed the cladding at the fiber end over a length of approximately 10 cm. We believe that the fiber can still withstand regular handling in a laboratory.

Figure 3 shows the end of the MFP. The sample (here a commercial photoresistor) is illuminated with red light. The MFP model P450A can host samples mounted on a resistivity board, and it includes a thermometer close to the sample, which records the temperatures during a measurement run.

Figure 4 shows the top end of the MFP insert in the PPMS.

Figure 2: Fiber installed to MFP. Fiber outlet inside MFP case: polished circular surface perpendicular to fiber. Fiber outlet protected and centered by an anti-magnetic alloy.

Figure 3: Sample part of the MFP with a commercial photoresistor.

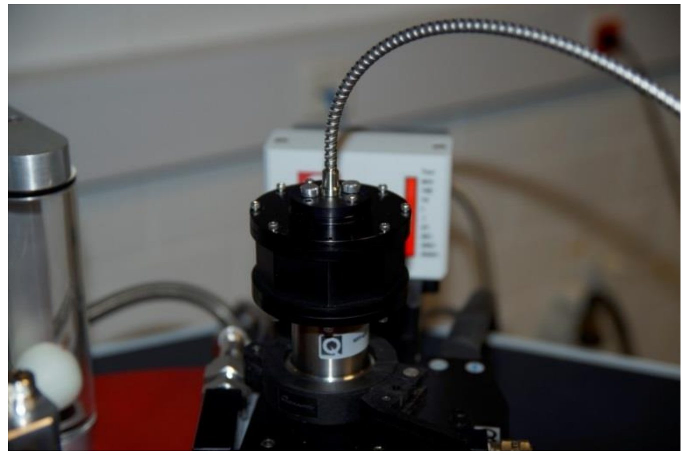

Figure 4: MFP with fiber mounted in the PPMS. The fiber goes through the center feedthrough and provides a gas-tight solution.

Figure 5: The fiber end is just screwed to the output of the monochromator. This monochromator has a variable slit width. For easy handling and more robustness, a commercial unit will have a slit with a fixed width.

Computer control of the MLS

The monochromator is motorized and computer-controlled via USB. For example, the user selects a wavelength and the monochromator positions the right grating and filter for this particular wavelength. An easy and direct way to do this is an intuitive dialog window. The monochromator may also be controlled from a PPMS MultiVu sequence by using a dynamic linked library (DLL). Win Wrap Basic (WWB) programming is used for this, which is embedded in MultiVu as a scripting environment. A special script was used for the measurements discussed below. Figure 6 shows the dialog generated by this example. The user can choose one of the following methods in combination with the PPMS's DC resistivity option (Model P400): scan time, scan temperature, scan field, scan wavelength, setting a single wavelength or a dark position (called "blank" in the mask). The scans done by this tool are single direction scans. Multiple scans will provide data in multiple data files.

As an alternative to probing the electro-transport properties via the DC resistivity option, we have also developed a setup for using a GPIB-operated third-party measurement bridge with the PPMS.

The software package also includes a set of several templates, which can be used for MultiVu scripting. Figure 7 shows an example. Using these templates provides the operator a more flexible handling of the monochromator compared with the above ready-to-use script.

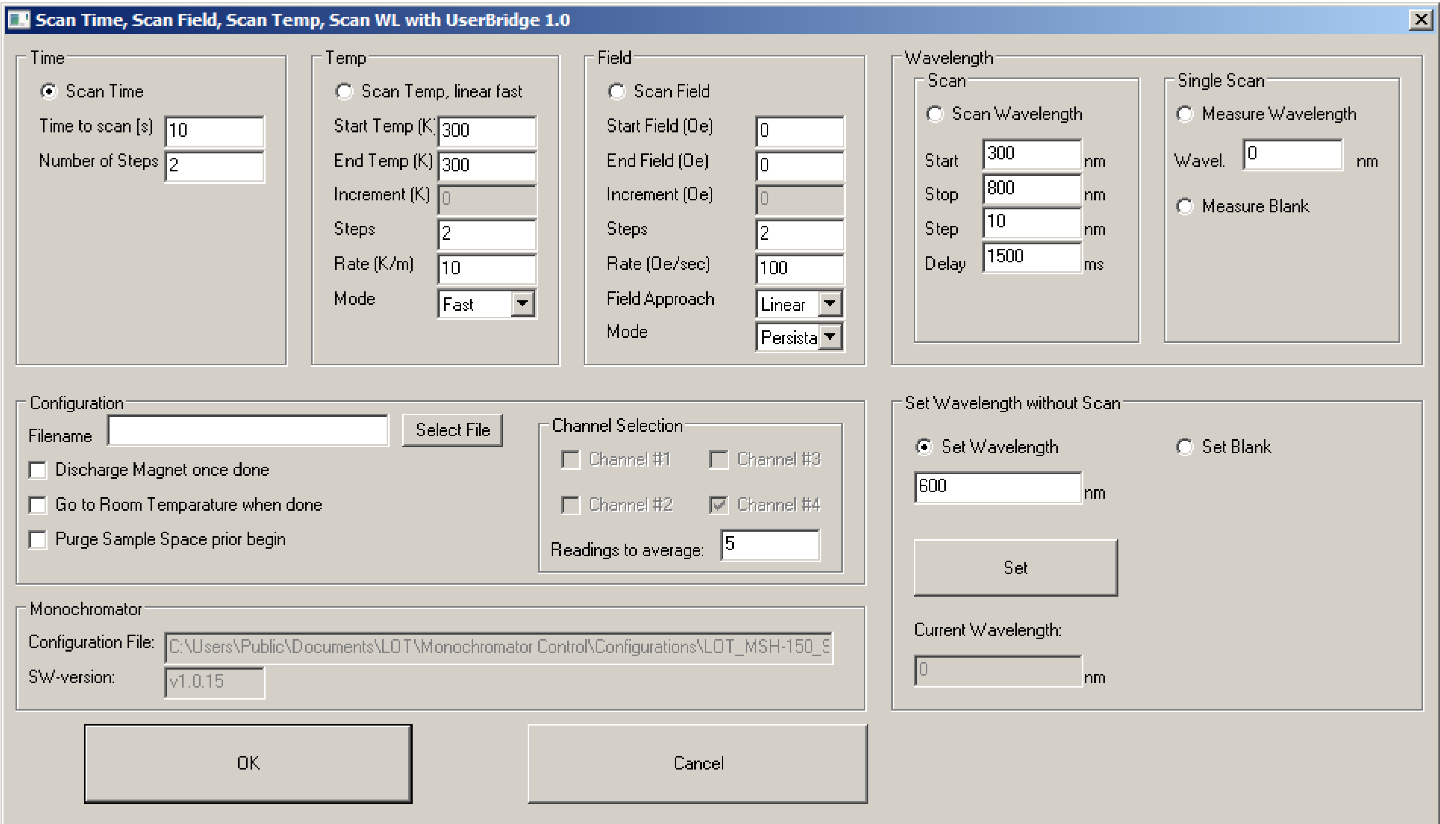

Figure 6: Window for MLS control, which can be used after starting the script.

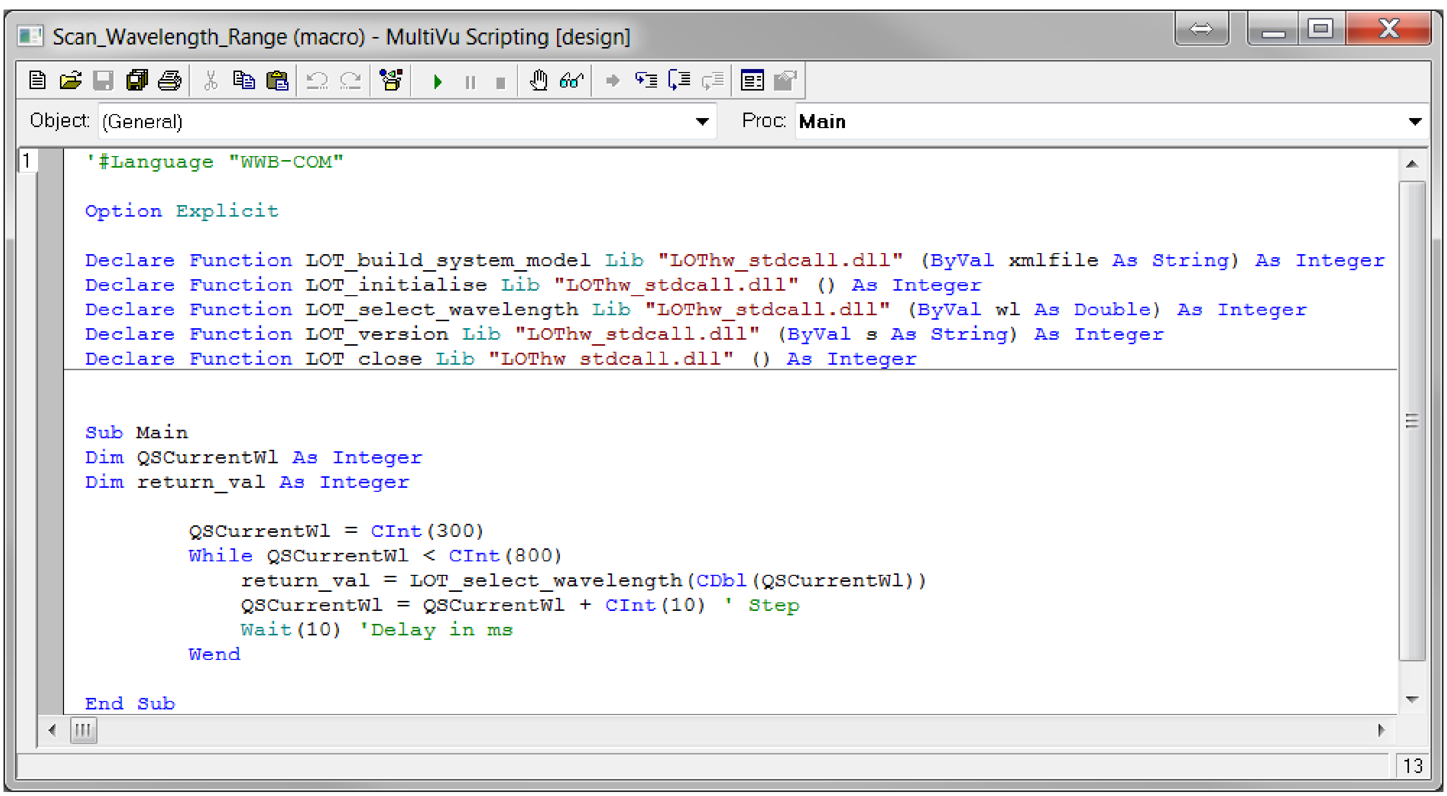

Figure 7: Example of a short MultiVu script, which scans the wavelength from 300 nm to 800 nm with 10 nm step width.

Spectral characterization of the MLS

The intensity of the light output was recorded with a spectroradiometer from Spectral Evolution (Model SR1900). This unit has two photodiode arrays. The silicon-based array covers the range from 350 nm to 1000 nm while the Indium-Gallium-Arsenid (InGaAs)-based array is used for the range from 1000 nm to 1900 nm. The complete spectral range is taken with one measurement. Figure 8 shows the relative intensity over the complete VIS and NIR range. Data points were taken by stepping the MLS from 390 nm with a 30 nm step size. The maximum intensity in the VIS range was observed at 660 nm. The spectral characteristics of the MLS set to 690 nm are shown in Figure 9. The bandwidth of this emission is approximately 10 nm (FWHM).

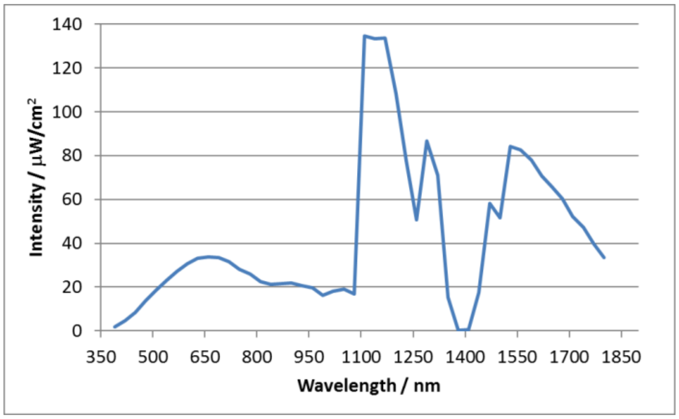

Figure 8: Relative intensity of the used setup. The large absorption around 1400 nm is due to absorptions of the UV/VIS fiber ("water band"). The significant intensity increase at ~1100 nm is due to the different parameters of the grating (lower line density) used for higher wavelengths.

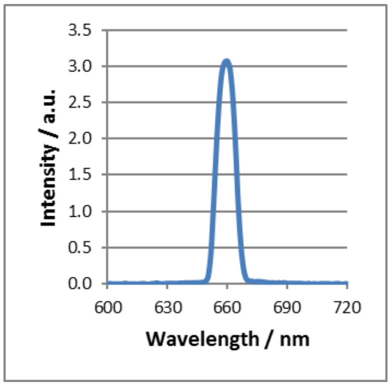

Figure 9: Emission with MLS set to 660 nm.

System configuration

Quantum Design/LOT offer the PPMS Fiber Probe as an integrated system. The default configuration for the PPMS Fiber Probe is given in Table 1 below. The MLS has a modular setup and can be customized. Please contact LOT or your local QD representative in case that a different configuration is requested.

Table 1: Default PPMS Fiber Probe configuration

Lamp	100 W halogen
Monochromator	Two gratings each with 600 lines/mm Expected bandwidth: ~20-25 nm Fixed slit widths
Fiber type	VIS/NIR type
Sample mount	Rotator board (model P103A). Supports two independent four-wire measurements and includes thermometer (see also Figure 3)

Measurement example

To demonstrate the capability of our setup for photoconductivity measurements, we have chosen as sample a commercial photoresistor based on CdS. The photoresistor specifications are summarized in Appendix C.

"On/Off" test

To show that the selected sample is functional, we have measured the resistivity while the light was manually switched on and off at a sample temperature of 300 K. A standard bulb was placed close to the fiber end so the light can be considered white. Figure 10 shows the diagram. In the dark position, the DC resistivity option cannot measure the resistivity of our sample, so the real resistance is going to be much higher than the 150 kOhm given in the sample's specification. According to the capabilities of our DC bridge, the real (dark) resistivity is greater than 40 MOhm.

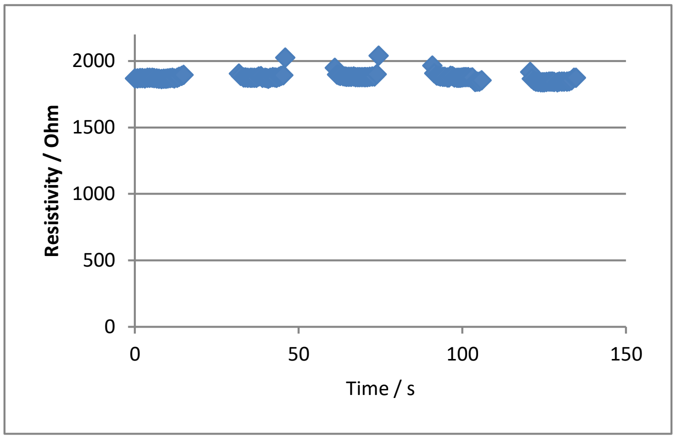

Figure 10: Switching between "light on" and "light off" at 300 K. The resistivity in the dark is out of the range of the used bridge.

Wavelength scan at 300 K

As next measurement, we performed a wavelength scan at 300 K. Resistance was recorded while the MLS scanned the wavelengths with 10 nm step sizes in the VIS/NIR range. The lowest resistance with 1.46 kOhm was observed at 580 nm. To scale the data point to the light intensity, the resistivity values were converted to the electrical conductance, which was then divided by the intensity measurements (see also Figure 8). Figure 11 shows the graph of the scaled wavelength scan.

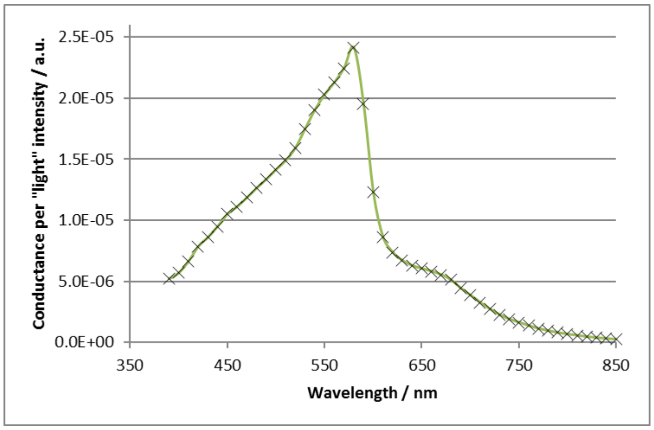

Figure 11: Relative electrical conductance of a commercial photoresistor based on CdS at 300 K as function of the illuminated center wavelengths.

The strength of the PPMS is that the system and sample environment parameters can be controlled through the software. This has been used to performed resistivity measurements at a variety of temperatures: 200 K, 100 K, 50 K, 20 K, 10 K, and 5 K. Figure 12 shows these wavelength scans. For moderate temperature reduction, the minimum of this curve is even lower compared with the 300 K measurement, before it increases significantly. The closer the temperature comes to zero, the higher is the resistivity. We expect that a semi-conductor will be perfectly isolating when it is at 0 K. The data in Figure 12 were not scaled on the light intensity as in Figure 11. However, the minimum in the resistivity curve is clearly visible. This minimum shifts towards lower wavelengths (equal to higher energies), when going down in temperature. It is 580 nm at 300 K, 570 nm at 200 K, 560 nm at 100 K and 50 K, and 550 nm at 20K, 10K, and 5 K. Another interesting fact is that some of the curves cross each other and not only shift towards higher resistivity values, which shall result from changes of the electronic states population at different temperatures. Since we do not know the exact material of our sample (especially of any potential dopants) and thus do not have any literature values for comparison, we did not make further evaluations.

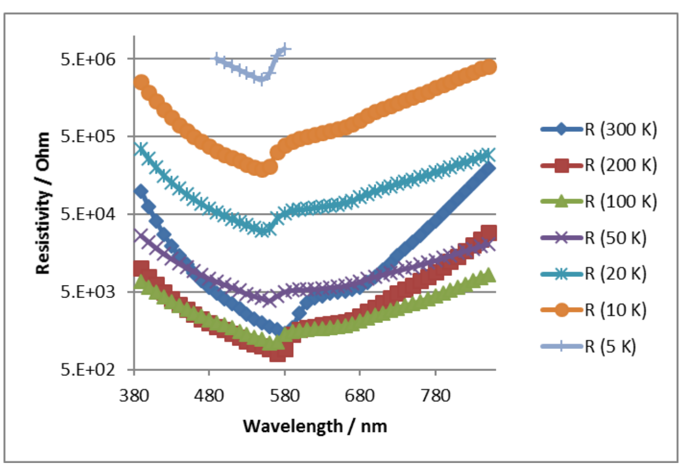

Figure 12: Wavelength scans at different temperatures.

Temperature scan

As an example of a temperature scan, the photoresistor was illuminated with 600 nm light while we measured the resistivity from 300 K to 2 K. Figure 13a shows the conductance as function of temperature, and Figure 13b has a logarithmic conductance versus 1000/T (assuming an Arrhenius type behavior). The maximal conductance was observed at 133 K. We also set up a temperature scan at a dark position, but the DC option could not detect any resistivity values (>40 MOhm) at any temperature.

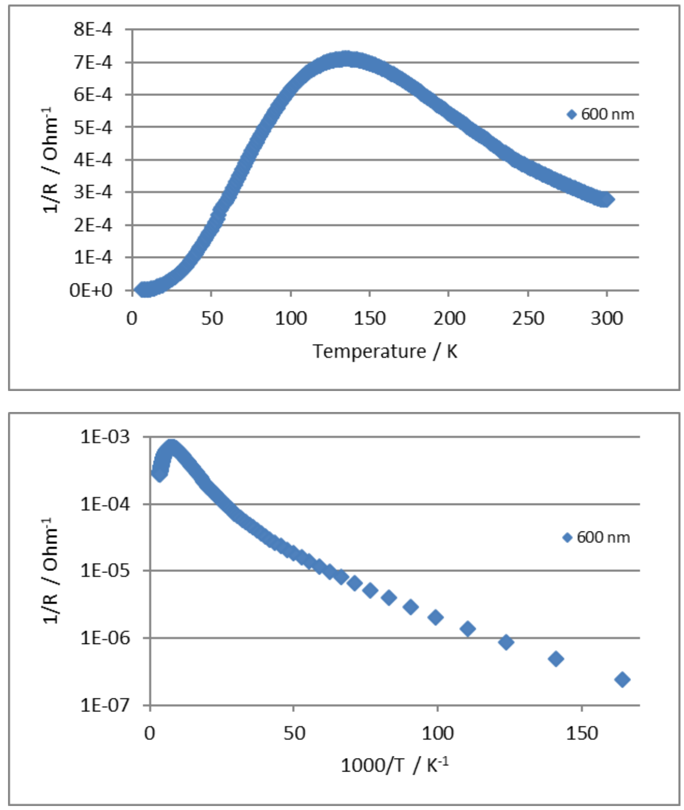

Figure 13a and 13b: Temperature dependence of the conductance with 600 nm illumination

Appendix A: characteristics of UV/VIS and VIS/NIR fiber

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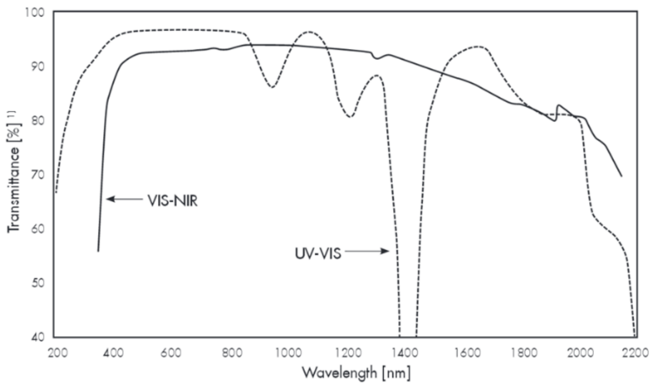

Figure 14: Comparison of the transmissions of the UV/VIS and the VIS/NIR fiber. The VIS/NIR fiber is a better choice if an illumination >1200 nm is desired.

Appendix B: theory

Photoconductivity describes the phenomenon that a semiconductor becomes more electrically conductive when illuminated by light of a certain wavelength (VIS, UV, IR etc.). The absorbed light generates charge carriers (electron and hole pairs). Well-known materials showing photoconductivity are cadmium sulfide (CdS), cadmium selenide (CdSe), lead sulfide (PbS), molybdenum disulfide (MoS2), selenium (Se), silicon (Si) and others. Doping a semiconductor can increase the photoconductive effect by generating charge carriers through the doping material, but it can also have a quenching effect. Panda et al. have investigated CdS doped with copper and describe an increase at a small doping concentration, but a decrease of the photocurrent at higher concentrations.[1]

The opposite effect, namely that the electrical conductivity decreases while the sample is illuminated has also been observed and is called negative photoconductivity (for example seen on graphene). Our test sample is a commercial photoresistor, or light dependent resistor (LDR), based on CdS. In CdS, the mobility of the electrons is much greater than the mobility of the holes. This means that the electric current is mainly dominated by the electron (n-type). CdS is known to have a slow reaction when the light is switched on and off. Even at room temperature, the material is not suitable for the detection or observation of very fast on/off processes, with very fast meaning fractions of a second. The kinetic of the electron-hole recombination shifts towards larger time scales at lower temperatures. The effect, that a sample still shows photo-current after switching the illumination off, is named as transient photoconductivity.

Figure 14 shows an example of the commercial photoresistor taken at a temperature of 40 K (please note that the conductivity in the dark at 40 K with the used bridge shall be zero).

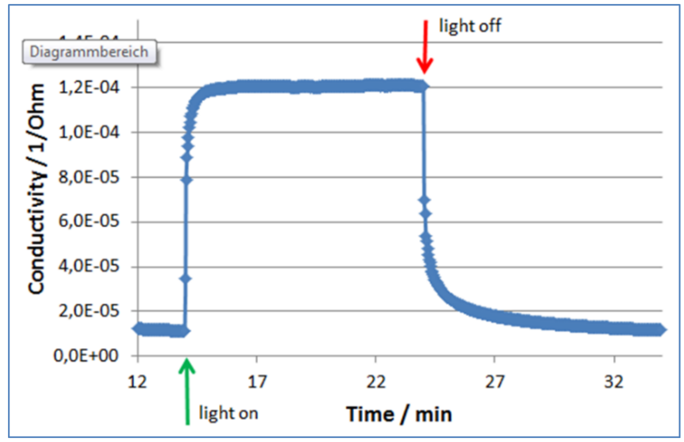

Figure 15: Kinetic effect and transient photocurrent at 40 K

The energy of the photon which generates the electron-hole pair should be at least be as high as the band gap. CdS has a band gap of 2.42 eV at 300 K, which corresponds to a wavelength of 512 nm. Studies of the wavelength-dependent photoconductivity can provide information about the band structure and forbidden regions.

The interaction of semiconductors, light and electric current is of general interest. Apart from the sciences, there are many commercial applications these materials are used for. For example, photovoltaics, LEDs, optical detectors, sensors and more. Even if most of these applications are at or close to room temperature, analyses at a wider temperature range or in depence of excited wavelengths provides a deeper understanding and should be part of the complete characterization of the semiconductor material.

Appendix C: commercial photoresistor

Table 2: Specification summary for the used commercial photoresistor as provided from the manufacturer's datasheet.

Model	Excelitas M 9960 11A
Dimensions	Flat design with approximately 5 mm x 5 mm area. Encapsulated in epoxy
Operating temperature range	-20°C to +70°C
R (10 lux)	1.5 to 5.0 kOhm
R (100 lux)	0.7 kOhm (typ.)
R (dark, after 5 s)	min. 150 kOhm
λPEAK	600 nm

[1] R. Panda et al., Appl. Surf. Sci. 258 (2012), 5086