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High-Speed MWIR Upconversion Spectroscopy

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Abstract

Infrared spectroscopy is a key technology for process analytical applications. Although stronger spectroscopic features are found in the mid-infrared at wavelengths beyond 3 µm, the near-infrared range is often favored due to better detector performance. In this work, continuous-wave nonlinear-optical upconversion is used to transfer signals from the mid-wave infrared to the near-infrared range where high-speed and high-sensitivity detectors are readily available. A grating spectrometer device for the mid-wave range, based on nonlinear-optical upconversion is presented as a promising tool for high-speed mid-infrared spectroscopy. The system is characterized for resolution and sensitivity. The high spectra acquisition rate allows combustion analysis in the wavelength range from 3.7 to 4.7 µm with outstanding time resolution.

Key words: Nonlinear optics, upconversion, spectrometer, infrared spectroscopy, combustion analysis

Infrared Detection by Upconversion

In process analytics, infrared spectroscopy is used for species detection, material and composition analysis, sorting and reaction monitoring. Many applications are covered by near-infrared (NIR) silicon- or indium-gallium-arsenide-(InGaAs)-based array spectrometers. At wavelengths longer than 3 µm, the strong fundamental vibrational transitions would allow higher sensitivity for spectroscopic measurements. Photon detectors for the mid-wave-infrared (MWIR) range of 3-5 µm, even when cryogenically cooled, are however limited in their sensitivity by their higher dark noise [1]. As a result, mid-infrared spectroscopy often relies on Fourier transform spectrometers (FTIR) or quantum- or interband-cascade laser (QCL/ICL) spectrometers where the spectral information is obtained by tuning of the laser. For analytics of highly dynamic processes over a broad wavelength range however, a grating-based array spectrometer would be a desirable option, since spectra can be acquired in a single shot.

In this work, a spectrometer system is presented, that relies on nonlinear-optical upconversion. Upconversion as a mechanism for infrared detection has been thoroughly discussed [2-4], also specifically as a tool for infrared spectroscopy [3, 5, 6]. Upconversion of infrared signals is achieved by mixing with a continuous-wave 1064-nm pump laser in a periodically poled lithium niobate crystal (PPLN) for sum frequency generation. Thus, light is transferred from the MWIR range to wavelengths between 780 and 880 nm, and can then readily be detected with a silicon-based array spectrometer. Since silicon array detectors are an abundant and matured technology, this approach enables construction of a sensitive and versatile grating spectrometer system for the MWIR.

The Upconverter Spectrometer System

In the presented system, a combination of an upconverter module and a subsequent high resolution grating spectrometer is used in order to acquire spectra from the MWIR on a conventional silicon sCMOS camera. The basic design is similar to the setup of a pulsed-laser upconversion spectrometer as presented in [3]. A schematic drawing of the system is shown in Fig. 1. For flexible light collection, the system is coupled to a ZrF_4 infrared fiber. From the fiber, MWIR light is focused to the center of the PPLN nonlinear crystal. The crystal is 20 mm long and has a 2x2 mm² entrance aperture. It is held in a temperature stabilized mount and is centered in one focus of a passive optical cavity. Locking electronics keep the cavity in resonance to the 1064 nm pump laser. An input laser power of 5 W is enhanced in the cavity to about 500 W of circulating continuous-wave power, providing high intensity inside the crystal for the conversion.

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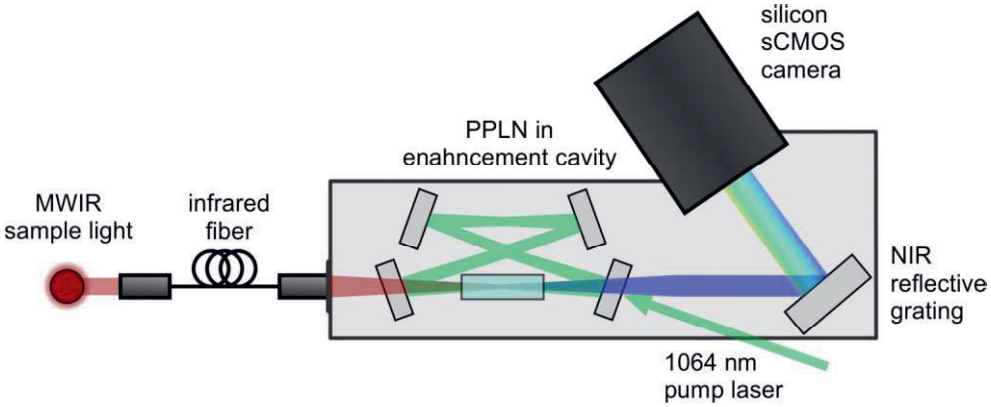

Fig. 1: Schematic drawing of the MWIR upconverter spectrometer system.

The upconverted light is collimated to a 1800-line-per-mm NIR grating. The diffracted spectrum is then imaged to the NIR camera. On the silicon-array camera used here, the height of the focused spectrum corresponds to 12 lines of pixels. These 12 lines can be read with a rate of 6500 frames per second. The camera was chosen for a good overall combination of sensitivity, dynamic range and speed, but event higher frame rates are possible with suitable array detectors.

The wavelength and intensity calibration of the spectrometer system was achieved with measurements of a blackbody radiator (Fig. 2a), as well as transmission measurements of various gas and polymer samples. For the gas samples, simulations based on HITRAN [7] were used as reference. For the polymer samples, FTIR measurements served as a reference. From a transmission measurement of CO_2 diluted in air compared to a corresponding HITRAN simulation (Fig. 2b), a wavelength resolution of approximately 2.3 nm (1.3 cm^{-1}) at 4.4 µm was estimated. This resolution is high enough to resolve single ro-vibrational gas absorption lines, remarkable for a mid-infrared array spectrometer.

The noise equivalent power (NEP) of the system was determined by comparing the measured blackbody emission spectra with the dark measurements. Here, the NEP refers to radiation power at the fiber entrance per wavelength bin, at an average bin width of 0.56 nm in the observed mid-infrared spectrum. Due to spectrally varying conversion efficiency and performance of the optical components, the NEP of the system varies across spectral range by a factor of 8, approximately. Nevertheless, a NEP of few pW $Hz^{-1/2}$ is achieved over the full sensitive range, spanning almost one micrometer between 3.7 µm and 4.7 µm (Fig. 2c). In the peak sensitivity area around 3.8 µm, the NEP lies below the 1 pW $Hz^{-1/2}$ level. The high sensitivity achieved by the setup is mainly due to the low-noise detection with the silicon-based detector, enabled by the upconversion. In the current setup, the sensitivity is limited by the system's quantum efficiency, i.e. the ratio of detected photons to photons at the fiber entrance. The efficiency lies around 10^{-4}, which is in part a consequence of the targeted broadband conversion scheme.

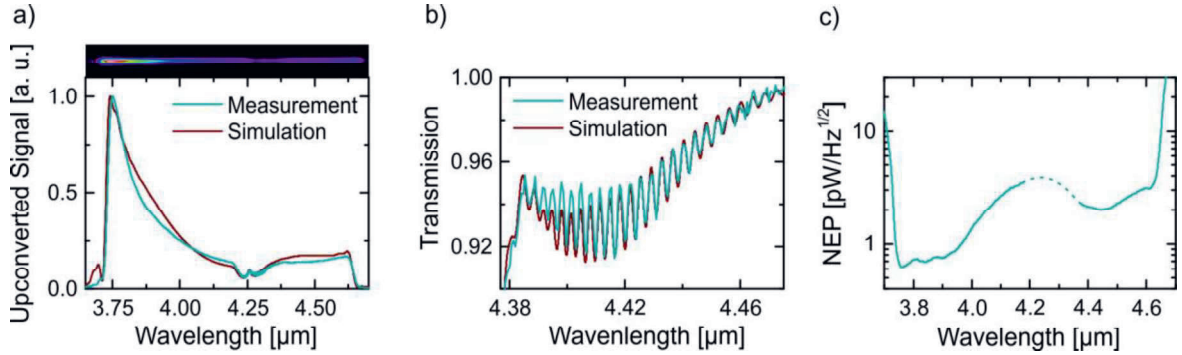

Fig. 2: a) Raw signal of a blackbody radiator at 1000 °C (top) and the measured raw spectrum, compared to a theoretical prediction of the signal. b) Zoom into a transmission measurement of CO_2 diluted in air, compared to a theoretical simulation with assumed instrument function of 1.3 cm^{-1}, demonstrating the wavelength resolving power of the spectrometer. c) Noise equivalent power of the system over the sensitive range. The dashed region is an interpolation across the absorption region of CO_2 in ambient air, corresponding to the case of a nitrogen purged setup.

The efficiency will be subject to further development of the system. While the wavelength range examined here is well suited to monitor gaseous combustion products, adaptations of the system will allow one to move the covered wavelength range to the hydrocarbon band around 3.3 µm.

High-Speed MWIR Combustion Analysis

Combustion of fuels is a prominent example of a highly dynamic multi-component process. The converter spectrometer system was utilized to study flame emission spectra during the combustion of a pyrotechnic airbag inflator. For this purpose, the fiber optic of the spectrometer was coupled to a gas exit nozzle of an inflator capsule. A sapphire window protected the fiber end from contact with the hot combustion gases (Fig. 3).

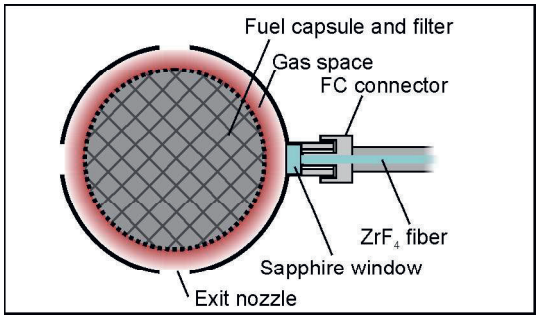

Fig. 3: Coupling of the optics to the combustion gases inside the pyrotechnic airbag inflator.

In the measurement, the maximum acquisition rate of 6500 spectra per second was used, with an exposure time of 150 µs per frame. The camera was synchronized to the ignition trigger. The acquired series of spectra show the dynamics of the combustion (Fig. 4). Distinct gas emission spectra can be observed, originating from hot CO_2 between 4.15 and 4.4 µm and other gaseous components at longer wavelengths. Observable effects are the re-absorption of emitted radiation by cooler CO_2 accumulating over time in the field of view as well as an increasing blackbody background at wavelengths < 4.1 µm from the capsule heating up.

Spectroscopic analysis as shown here has the potential to aid understanding of the non-equilibrium combustion kinetics, due to the high achievable time resolution.

Conclusion

By using continuous-wave nonlinear-optical upconversion, a spectrometer system was designed, capable of acquiring spectra from the mid-wave infrared range, spanning a range of one micrometer, on low-dark-noise silicon devices. This allows for sensitive spectroscopic measurements, with noise equivalent powers in the range of 1 pW $Hz^{-1/2}$ and below.

In the current setup, spectra can be acquired at a maximum rate of 6500 spectra per second. This was used for a high-time-resolution analysis of the combustion process in a pyrotechnic airbag inflator module.

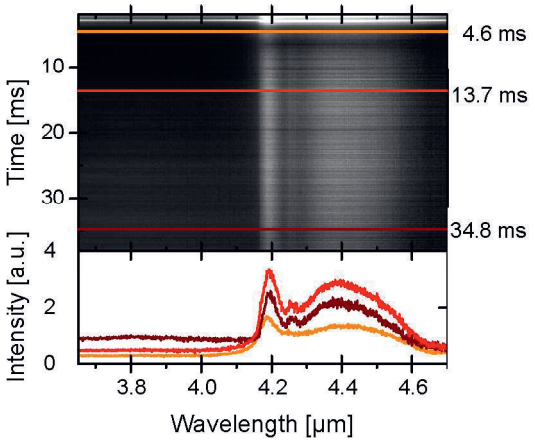

Fig. 4: Flame emission spectra taken during combustion in the pyrotechnic airbag inflator. The full spectra time series (top) is taken with a time resolution of 150 µs. At the bottom, three single spectra corresponding to the marked times after the ignition signal show the spectral dynamics.

The possibility to use silicon-based array detectors enables convenient and flexible design of future systems. Further increase of the system's sensitivity by improvement of the conversion process and the adaptation to other wavelength ranges are the main tasks of on-going development.

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