HYDROPTICS Press Release

Dual-Comb spectroscopy for oil detection

Background/Motivation:

Detection of oil with single color Distributed Feedback Quantum Cascade Laser (DFB QCL) laser requires a reference sample and/or an auxiliary laser with different color for background determination. With external cavity QCLs this issue is resolved, since the laser wavelength can be tuned by several hundreds of inverse cm, however, the simultaneous detection of the background and sample is not possible. These limitations of single color DFB QCLs and external cavity QCLs for measurements of oil in water content are resolved by employing dual frequency comb spectroscopy, which enables broad band spectral measurements without the complicated mechanical system employed in external cavity QCLs. Until a few years ago, frequency comb sources in the mid-infrared spectral region were bulky and delicate contraptions, a long way from the small and robust instruments desired in process analytical applications. Recently, a novel kind of mid-infrared frequency comb source based on quantum cascade lasers was demonstrated [1]. A quantum cascade laser frequency comb comprises all important features of a mid-infrared frequency comb on a single semiconductor chip of a few square-millimeters in size. This ground-breaking invention opens the way for integration of frequency comb spectroscopy in laboratory and also everyday life applications.

Dual frequency comb spectroscopy is a seminal new way of measuring optical spectra. Its concept and the associated innovation potential were honored with the physics Nobel Prize in 2005. The operation principle of dual-comb spectroscopy is illustrated in Fig. 1: two frequency comb laser sources are superimposed, and the spectrum is measured on a single detector. Both light sources have a set of equidistant emission lines, though the distance between the lines is different for the two sources (see right hand side of Fig. 1(b)). The mixing of the two waves on the detector produces a multi heterodyne signal, where every pair of optical lines produces a signal at a distinct frequency in the radio frequency domain. These radio frequency signals can be measured by conventional electronics.

Currently, QCL dual-comb spectroscopy is commercially available as a table-top instrument for research applications. It is optimized for high-speed measurements with microsecond time resolution and below 10^{-4} cm^{-1} spectral resolution [2]. As such, it was successfully employed for numerous applications such as protein dynamics studies, combustion analysis, or remote explosives detection [3]. Its commercial success mostly builds on its exceptionally low noise levels in short measurement times.

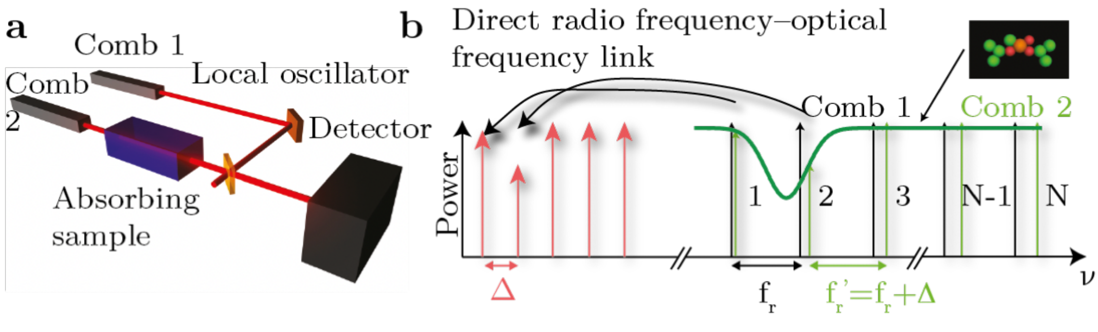
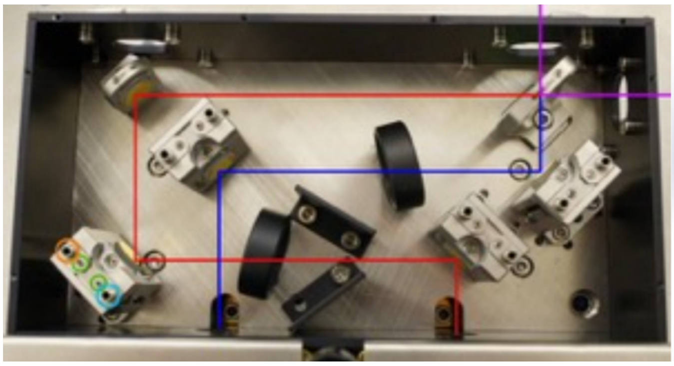


Fig. 1: a) dual comb setup schematic with signal and local oscillator comb mixing on a single detector element. b) overlapping optical spectra of two combs (black and green) and down converted multi-heterodyne in the RF domain (red), which can be evaluated with electronic equipment. Absorbed intensity in the comb lines translates to reduced intensity of the corresponding beat note.

Developments within HYDROPTICS:

With the developments in Hydroptics, we want to unlock the process analytical technology market for QCL dual comb spectroscopy. In addition to the fast, low noise measurements of the technique, we mostly build on three other inherent advantages of frequency comb spectroscopy: the exceptional robustness of the measurement principle, reduced fingerprint of the device, and the potential of low-cost production. Both are to a good part caused by the absence of moving mechanical elements in dual-comb spectroscopy. While the current dual-comb spectrometer already exhibits great mechanical stability, some major development steps are planned to reduce the costs and footprint of dual-comb spectroscopy.

During the execution of Hydroptics, the free-space optical beam combining of current day dual-comb spectrometers will be replaced by an integrated optical circuit. State of the art beam combining in dual comb spectroscopy relies on macroscopic plate beam splitters, where the two laser beams have to hit the beam splitter surface at exactly the same place under the correct angle. To achieve that, a number of optical elements with each a high-performance opto-mechanical mount is required, as can be seen in the photograph of IRSWEEP's current beam combiner module in the Fig. 2. In Hydroptics we will go beyond that state-of-the-art, by replacing the complicated free-space optical path by an integrated optical circuit as sketched on the right-hand side of the Fig. 2. The integrated optical circuits are realized using Si fabrication technology, making its fabrication precise, with high yield and low cost when produced in volume.



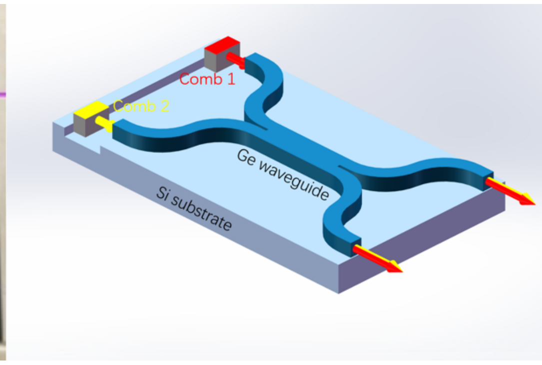


Fig. 2 Dual-comb beam combiner. Left hand side: free-space beam combiner as currently implemented at IRsweep's spectrometer. Right hand side: sketch of the integrated beam combiner with the QCL's directly coupled to Ge on Si waveguides.

Another major improvement developed within Hydroptics is a drastic reduction of the sampling rate required to acquire the multi-heterodyne signals. Current state of the art quantum cascade laser dual frequency comb spectroscopy employs free running frequency combs. Jitter in the frequency comb lines lead to a broadening of the detected heterodyne lines, resulting in a line width of 1-2 MHz. To properly resolve adjacent heterodyne lines, a spacing of the lines of > 2 MHz is required. With several hundred wavelengths detected in parallel, this sums up to several hundreds of MHz (up to 1 GHz) detection bandwidth. The high-speed digitizer acquiring 2 GS/sec is one of the main price drivers of the current dual-comb system. To reduce the requirements of the high-speed sampling, we propose in Hydroptics to implement a frequency stabilization of the frequency comb sources.

The lines of a frequency comb can be characterized by two frequencies, the repetition frequency f_{rep} and the carrier envelope offset frequency f_{CEO} and full stabilization requires the stabilization of both. The most widely known technique for frequency stabilization of frequency combs is the f-to-2f stabilization techniques [4]. However, it requires an octave spanning operation, which is difficult to be achieved with semiconductor lasers. For Hydroptics, we employ an alternative approach, which relies on the relative stabilization of one comb onto the other. Absolute referencing onto absorption lines will be done during the data analysis.

Recent findings revealed that f_{rep} can be locked to an external low-noise electronic oscillator, while maintaining frequency comb operation [5]. This technique increases the range of comb operation and its robustness against environmental influences, such as temperature fluctuations, laser current noise or vibration induced optical feedback. Most relevant for Hydroptics is the fact that injection locking provides the missing control knob to independently stabilize both f_{rep} and f_{CEO}. As f_{rep} remains fixed, the laser current only impacts f_{CEO} and hence can be used for stabilization within a control loop that acts onto the laser current. In Hydropics we will implement this stabilization technique in a mid-infrared QCL dual-comb spectrometer together with subsampling techniques. This will reduce the sampling rate from currently 2 GS/sec to 20 MS/sec. By this, it will cut component costs of the dual-comb system in the 5-digit range.

References

[1] Hugi et al. “Mid-infrared frequency comb based on a quantum cascade laser” Nature (2012) 492, 229–233.

[2] See IRis-F1 datasheet: http://cloudfront.irsweep.com/resources/IRsweep_IRisF1.pdf

[3] Klocke et al., Anal Chem 17, 10494 (2018); (b) Pinkowski et al., arXiv:1903.07578 (2019); (c) Hensley et al., Proc. SPIE 1063820 (2018).

[4] Apolonski et al. Phys. Rev. Lett. 85,740 (2000)

[5] Hillbrand et al., Nature Phot. 13, 101 (2019)