SUCCESS STORY

CHASE
Chemical Systems Engineering

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QCL-Sensorplattform; installiert in einem Industrierack. (© CHASE/Moser)

QUANTUM CASCADE LASER SENSOR FOR SULFUR SPECIES DETECTION IN PETROCHEMICAL PROCESS STREAMS

THE DEVELOPED PROTOTYPE TECHNOLOGY RESULTED IN VERY LOW DETECTION LIMITS FOR SULFUR BASED COMPOUNDS

The sensitive and selective detection of gaseous sulfur species with the emphasis on hydrogen sulfide (H_2S), carbonyl sulfide (COS) and methyl mercaptan (CH_3SH) down to sub-ppmv concentrations plays a crucial role across a wide range of petrochemical applications including production control, quality assurance or personal safety monitoring purposes. The wide occurrence of sulfur species in these processes and due to their negative impact on process stability and product quality, the concentrations need to be tightly monitored in the sub-ppmv range for H_2S and CH_3SH, whereas sensitivity of ~50 ppbv for COS quantification is demanded.

Despite a variety of online monitoring options for gaseous sulfur species, their quantitative reliable determination still remains a challenge in the field of chemical sensors.

As a consequence, a sensitive, selective and industrial fit gas sensor based on second harmonic wavelength modulation spectroscopy (2*f*-WMS) employing three continuous wave distributed feedback quantum cascade lasers (CW-DFB-QCL) was developed for detecting gaseous sulfur species at sub-ppmv levels in petrochemical process gas streams at the project partner OMV.

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The laser radiation of the individual QCLs is frequency multiplexed, co-aligned and focused with beam splitters and plano-convex lens systems.

The combined laser beams are further split into a reference and signal path before being coupled into an astigmatic 76 m Herriott-type multipass cell and subsequently focused onto an optically immersed, thermoelectrically cooled HgCdTe detectors.

The reference path is equipped with methane (CH_4), H_2S and carbon monoxide (CO) reference gas cells for accurate wavelength calibration and laser drift compensation.

The detector signals are digitized, de-glitched and demodulated at the individual modulation frequencies and further processed using a self-developed field programmable gate array (FPGA) implemented lock-in amplifier. The pressure broadened spectra are subjected to a modulation index compensation method based on the zero-, second-, and fourth-order harmonics of WMS. Signal averaging over 10 sweeps results in a total response time of ~1 s.

In order to meet with on-site safety regulations, the three-channel QCL sensor platform is installed in an industry rack and equipped with the required safety infrastructure allowing a certified operation under ATEX/IECEx regulations for hazardous and explosive environments. The sensor rack combines a fully automated gas probing and conditioning system alongside with a purge and pressurization system with intrinsic safety electronic devices achieving a versatile explosion prevention and malfunction protection.

Quantitative measurements of H_2S, CH_3SH and COS were performed using calibration gas mixtures in nitrogen (N_2), methane (CH_4) and propene (C_3H_6) matrices in order to investigate the sensitivity and linear response of sensor system. The corresponding limits of detection (LOD) were assessed according to DIN 32645 and resulted in ~0.3 ppmv for H_2S, ~60 ppbv for CH_3SH and ~5 ppbv for COS respectively.

The spectral and chemical information retrieved with the sensor platform is crucial for gaining in-depth process understanding and to ensure optimum process operation conditions.

Furthermore, the developed prototype technology is being transferred to industry partner OMV further continued instrument testing and development.

Project coordination (Story)
Harald Moser, DI Dr.
Project Manager
CHASE GmbH
T +43 664 88498705
harald.moser@chasecenter.at

Competence Center CHASE GmbH
Altenbergerstrasse 69
4040 Linz
T +43 664 9658923
Patrick.pammer@chasecenter.at
www.chasecenter.at

Project partner

- OMV, Austria
- TU Wien, Austria

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