An Integrated IR-Sensor for Vibrational IR-Spectroscopy

Jürgen Kasberger

Research Center for Non Destructive Testing GmbH (RECENDT),
Hafenstraße 47-51, 4020 Linz, Austria

Introduction

In industry, a vast variety of processes can be optimized by means of online measurements of chemical properties of fluids in order to reduce costs of maintenance or increase productivity as well as production quality. Considering the vast verity of fluids present in industrial applications suggests the utilization of a vibrational spectroscopic method, which offers high selectivity as well as high sensitivity. As an alternative to commercial IR-spectrometers, which are in general expensive and bulky instruments, different components related to miniaturized spectrometers have been investigated in the recent years. In particular, a lot of work has been done in the field of the spectroscopic measurement method, where a number of publications are dedicated to absorption in the evanescent optical field. The most prominent representatives of optical waveguides are optical fibers, which have been investigated thoroughly (e.g., [1] – [3]). However, due to the geometrical dimensions of conventional fibers, the evanescent field is relatively small and hence a lot of effort has been done to improve the sensitivity, e.g., by means of tapered structures [4], [5]. To exploit the advantages of guided optical waves for measuring purposes, also photonic structures [6] – [9] have been considered.

Working with photonic elements (waveguides), which utilize the absorption in the evanescent field, requires coupling of an optical beam into the waveguide. The guided optical wave of an evanescent field sensor is often excited by means of (slanted) front end coupling (e.g., [6], [8]) or prism coupling [10]. For spectroscopic measurements usually either optical fibers in conjunction with an interferometer or recently quantum cascade lasers (QCL) [11] – [13] are deployed.

In addition to the evanescent field sensor element, a lot of work has also been subjected to miniaturize spectrometers. Most of the work is dedicated to miniaturize the dispersive element [14]. There have also been attempts to integrate a wavelength selective measurement in conjunction with an evanescent field sensor element [15], [16]. For example, in [15] the IR-radiation is excited by heating the suspended waveguide itself, which results in broadband IR-radiation guided in the waveguide. However, due to the applied band-pass filter, the setup is limited to a specific wavelength.

In contrast to [17], [18], where the dispersive features of gratings (couplers) are exploited as a sensing mechanism, we utilize the gratings as dispersive elements as well as for waveguide coupling. Thus, our approach to fully integrate an IR-absorption sensor is different from the work done so far focusing on specific features of single parts of a sensor system like, e.g., grating couplers.

Concept of a Fully Integrated IR-Absorption Sensor

We aim at a fully integrated absorption sensor, as sketched in Fig. 1, based on IR-absorption in the evanescent field of an integrated slab waveguide utilizing thermally generated and detected IR-radiation. For the proposed sensor system, the coupling by means of grating couplers is advantageous, since front end coupling is not suitable for the considered mono-mode waveguide of the proposed setup.

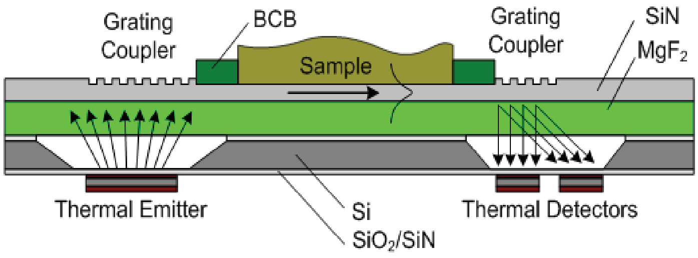

Fig. 1: Concept of a fully integrated sensor system for the mid IR-range. Two grating couplers are utilized (1) to couple broadband IR-radiation into the waveguide and (2) subsequently for spectral separation when coupling out of the waveguide.

The thermal radiation excitation and detection – influencing the design of the grating couplers – was selected due to the targeted mid-IR-region. Even though non-thermal elements exhibit outstanding features like, e.g., the high sensitivity of cryogenic detectors, those non-thermal elements are not suitable for integration into a micro-sensor due to the associated comparatively large dimensions.

In various targeted applications the measurement of the IR-absorption at a limited number of wavelengths is suitable to characterize a certain chemical property. For example, in order to reduce the interval of scheduled maintenance of lubrication oil, e.g., in combustion engines, the quality of the deteriorated oil has to be determined. The occurring oxidation of lubrication oil can be used as monitoring feature for the aging process. Since this oxidation can be characterized by measuring the IR-absorption at two distinct relatively broad wavelength ranges [19], low spectral resolution is required to determine the deterioration of lubrication oil. Therefore, the monitoring of lubrication oil [20], [21] was chosen as example application for the first experimental investigations.

Validation of the Waveguide as Transducer

In order to characterize the proposed mono-mode waveguide as an IR-absorption element, we have fabricated a waveguide structure including two grating couplers with a SiN-waveguide residing on a MgF_2-substrate. The concept of the fully integrated absorption sensor, as shown in Fig. 1, utilizes the angular coupling characteristics of grating couplers for the spectral separation of the measurement wavelength. To characterize the mere waveguide as absorption element, we have built up a measurement setup, where the spectral measurement is done with an IR-spectrometer [23]. For the characterization of the waveguide prototype, comprising the input grating coupler, the waveguide and the output grating coupler, the total transmission through the sample is measured for a certain fixed coupling angle.

In order to demonstrate the sensitivity of the propose mono-mode waveguide, measurements obtained with a commercial ATR-element (three reflections) are compared to those obtained with our waveguide structure, as shown in Fig. 2. For both evanescent field absorption elements (ATR and mono-mode waveguide) a reference measurement as well as the corresponding oil measurement, where the oil is loaded on top of the waveguide without changing the optical setup, are depicted. The decrease in transmission for the oil measurement compared to the corresponding reference measurement, which is only recognizable for the mono-mode waveguide (Fig. 1), confirms the fundamental sensitivity of the proposed mono-mode waveguide structure. With the obtained sensitivity, this measurement setup can also be applied for monitoring the deterioration of the investigated lubrication oil, as demonstrated in [23].

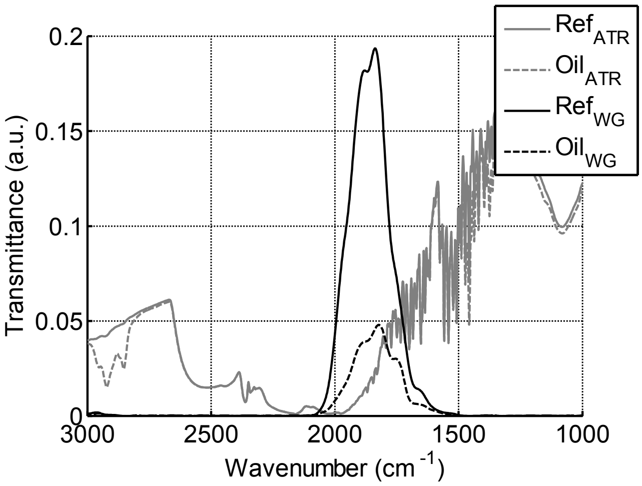

Fig. 2: Total transmission through the gratings and the waveguide of reference (i.e., no oil loaded on the waveguide) and oil measurements. Here a commercial ATR-element and the investigated waveguide prototypes are compared regarding the absorption in the evanescent field of devices.

Application to Deteriorated Lubrication Oil

In order to demonstrate that the concept, depicted in Fig. 1, can be utilized as oil aging sensor, the dispersive manner utilized for spectral separation has to be taken into account. Instead of using two IR-detectors, as shown in Fig. 1, a single element has been positioned with a motorized translation stage to scan the spectral range in the used lab setup.

For the application of the setup as oil aging sensor, the IR-emitter is positioned to obtain the maximum intensity at the IR-detector at about $\nu \approx 1800\text{cm}^{-1}$ ($\lambda = 5.5\mu\text{m}$), which corresponds to a coupling angle of about -10°. The center wavelength of 1800cm^{-1} is chosen due to the special characteristics of the considered lubrication oil, which does not change the absorption below this wavelength but has its typical oxidative absorption above this wavelength [19]. Once the IR-emitter is positioned, a reference measurement (without oil) is carried out followed by an oil sample measurement, where the optical setup is not changed between the reference and the oil measurement. To obtain a normalized transmission curve, characterizing the oil quality, it is necessary to relate the oil measurement to the reference measurement, which results in normalized transmission measurement with respect to the coupling angle. The normalized oil measurements are corrected to match in the region between -12° to -5°, as shown in Fig. 4, corresponding to the spectral region that is not affected by oil oxidation.

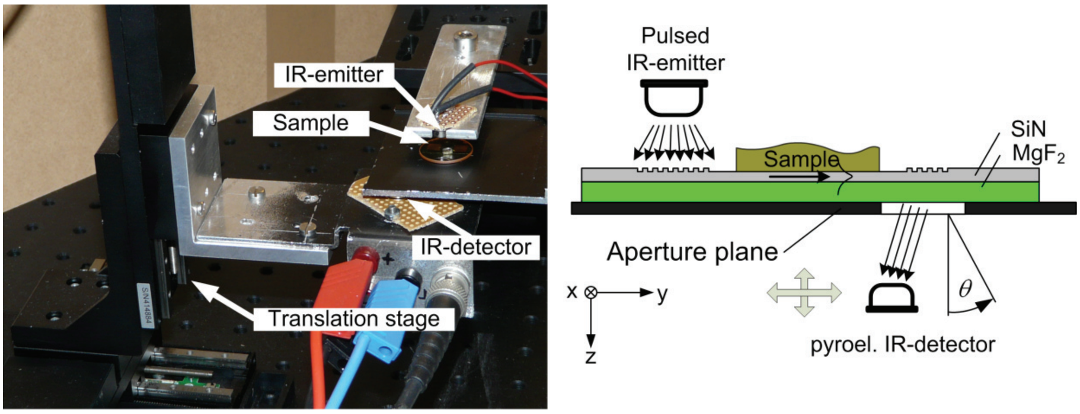

Fig. 3: Measurement setup including a motorized translation stage to investigate the spectral separation. The IR-emitter is positioned at the opposite side of the waveguide because this configuration makes the positioning easier. In a final setup both the emitter and the detector could be placed on the same side of the waveguide element.

Even though the aging can be clearly distinguished from the normalized transmission measurements of the differently aged oils, shown in Fig. 4, it is apparent that this prototype setup is not optimized regarding the noise behavior. Already a simple housing of the entire setup or the utilization of an IR-detector array, which would enable a simultaneous measurement of the considered coupling angles, would improve the signal to noise ration significantly. Even though the spectroscopic measurement, obtained by means of the dispersive features of a grating coupler, could be realized with an IR detector array, we have carried out our experiment with a mechanically positioned single element thermal detector in order to reduce the technical complexity.

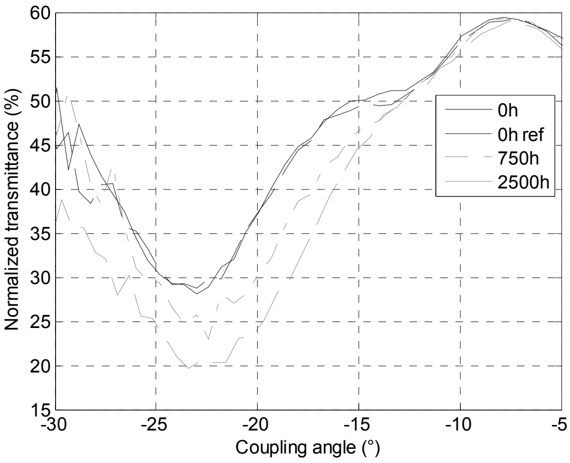

Fig. 4: Normalized (oil to reference measurements) transmission of different artificially aged oils versus coupling angle. The normalized transmission is shown twice (0 h and 0 h ref) in order to illustrate the degree of reproducibility.

Improved Sensor Performance

In the mid-IR-range the available power is limited, which results in high requirements with respect to the signal to noise ration. Since we utilize in the proposed fully integrated sensor thermal IR-detectors with less sensitivity than cryogenic cooled IR-detectors, we attempt to improve the sensitivity by means of exploiting the features of photonic structures, as shown in Fig. 5.

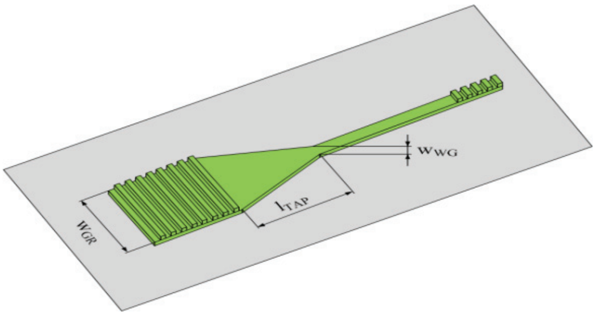

Fig. 5: Sketch of an integrated taper structure of a SiN-waveguide on an MgF_2 substrate. The insertion of the taper structure features an improved energy density resulting in a better signal to noise ratio.

A thermal IR-source simply represents a heated body emitting IR-radiation described by Planck's law. In case of a thermal emitter the power emitted by the surface is proportional to the surface area and the temperature. Thus, increasing the width (coupling area) of a grating coupler increases the energy coupled into the waveguide but not the energy density. A taper structure on the other hand increases the energy density (see Fig. 6) but not the total guided energy. In consequence, introducing a taper structure, as shown in Fig. 5, enables the implementation of a wide grating coupler – yielding high energy in a wide waveguide – in conjunction with a small waveguide guiding the IR radiation. This configuration can be utilized in combination with a small IR detector (array) obeying less noise than larger detectors.

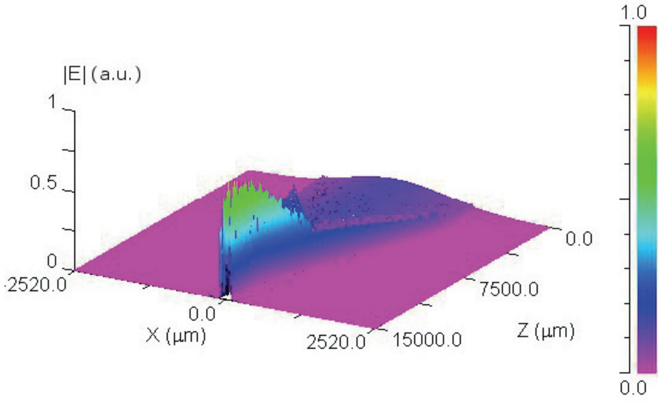

Fig. 6: Numerical simulation of the taper structure using the beam propagation method (BPM, RSoft). The calculations have been carried out in cooperation with the Center for Surface- and Nanoanalytics, Johannes Kepler University Linz.

Spectral Resolution

Even though the requirements regarding the spectral resolution are very low for the considered application regarding oil aging, it is of general interest to find out what spectral resolution can be achieved with the proposed sensor setup. Thus, in contrast to our previous work dealing with the sensor principle, we considered in [24] the achievable spectral resolution of the proposed concept, which represents a major requirement for further work. Using grating couplers, broadband IR-radiation can be coupled into and out of the waveguide, where the coupling angle determines the wavelength of the coupled radiation. Thus the couplers also facilitate separation of the prescribed wavelength. Besides the coupling efficiency of thermal radiation of the utilized grating couplers [22], the design of the mono-mode slab waveguide also affects the performance of the entire sensor system with respect to the signal to noise ratio (SNR) of the detected IR-radiation. However, considering the achievable spectral resolution requires a special consideration on the design of the grating coupler, which couples the IR-beam towards the IR-detector featuring the spectral separation. In order to confirm the numerical results obtained in [24], we also have carried out measurements by means of an experimental setup utilizing a single element detector and a simple 3 dimensional mechanical positioning setup.

Conclusion

Vibrational spectroscopy is a powerful method in characterizing chemical substances regarding their quantitative and qualitative composition. However, the application of vibrational spectroscopy in the different fields from process monitoring to lab applications requires a sophisticated interface between the IR radiation and the sample under investigation. Especially the emerging microsystems technology shows features beating the conventional measurement equipment. The investigations carried out so far, have shown promising theoretical and experimental result. However, there is still a lot of work to do in order to understand and exploit the full potential of photonic structures in vibrational spectroscopy.

Acknowledgment

This work has been supported by the European Regional Development Fund (EFRE) in the framework of the EU program Regio 13, the Austrian research funding association (FFG) under the scope of the COMET program within the research network “Process Analytical Chemistry (PAC)” and the federal state Upper Austria. The preliminary studies have been carried out at Integrated Microsystems Austria GmbH within the PhD study of the author at the Institute for Microelectronics and Microsensors, Johannes Kepler University Linz.

I would like to thank Ahmad Saeed (Center for Surface- and Nanoanalytics, Johannes Kepler University Linz) and Thomas Fromherz (Institute of Semiconductor and Solid State Physics, Johannes Kepler University Linz) and their colleagues for supporting the work related to microsystems technology. The fruitful cooperation and the thus facilitated access to the clean room facilities at the physics department enabled the sophisticated research in the field of microsystems technology.

References

[1] L. Küpper, J.V. Gulmine, P.R. Janissek, H.M. Heise, Attenuated total reflection infrared spectroscopy for micro-domain analysis of polyethylene samples after accelerated ageing within weathering chambers, Vib. Spectrosc. 43 (2004), pp.63-72.

[2] P. B. Tarsa, P. Rabionwitz, K.K. Lehmann, Evanescent field absorption in a passive optical fiber resonator using continuous-wave cavity ring-down spectroscopy, Chem. Phys. Letters 383 (2004), pp.297-303.

[3] B. Mizaikoff, K. Taga, R. Keller, Infrared fiber optic gas sensor for chlorofluorohydrocarbons, Vib. Spectrosc. 8 (1995), pp.103-108.

[4] M. Ahmad, Larry L. Hench, Effect of taper geometries and launch angle on evanescent wave penetration depth in optical fibers, Biosens. Bioelectron. 20 (2005), pp. 1312-1319.

[5] J. Keirsse, C. Boussard-Pl´edela, O. Lor´ealb, O. Sirec, B. Bureaua, P. Leroyerb, B. Turlinb, J. Lucasa, IR optical fiber sensor for biomedical applications, Vib. Spectrosc. 32 (2003),pp.23-32.

[6] G. Pandraud, T.M. Koster, C. Gui, M. Dijkstra, A. van den Berg, P.V. Lambeck, Evanescent wave sensing: new features for detection in small volumes, Sensor Actuators A 85 (2000), pp.158-162.

[7] M. Ishifuji, T. Suzuki, M. Mitsuishi, T. Miyashita, Molecular orientation of nonlinear optical polymer nanosheets on silica nanoparticle monolayer studied by optical waveguide spectroscopy, Thin Solid Films 518 (2009), pp.457-461.

[8] B. Mizaikoff, Mid-infrared evanescent wave sensors - a novel approach for subsea monitoring, Meas. Sci. Technol. 10 (1999), pp.1185-1194.

[9] G. Pandraud, P.J. French, P.M. Sarro, Fabrication and characteristics of a PECVD SiC evanescent wave optical sensor, Sens. Actuators A 142 (2008), pp.61-66.

[10] M. Zourob, S. Mohr, B.J. Treves Brown, P.R. Fielden, M. McDonnell, N.J. Goddard, The development of a metal clad leaky waveguide sensor for the detection of particles, Sens. Actuators B 90 (2003), pp.296-307.

[11] Ch. Charlton, A. Katzir, B. Mizaikoff, Infrared Evanescent Field Sensing with Quantum Cascade Lasers and Planar Silver Halide Waveguides, Anal. Chem. 77 (2005), pp.4398-4403.

[12] L. Hvozdara, N. Pennington, M. Kraft, M. Karlowatz, B. Mizaikoff, Quantum cascade lasers for mid-infrared spectroscopy, Vib. Spectrosc. 30 (2005), pp.53-58.

[13] Ch. Charlton, M. Giovannini, J. Faist, B. Mizaikoff, Fabrication and Characterization of Molecular Beam Epitaxy Grown Thin-Film GaAs Waveguides for Mid-Infrared evanescent Field Chemical Sensing, Anal. Chem. 78 (2006), pp.4224-4227.

[14] R. F. Wolffenbuttel, State-of-the-Art in Integrated Optical Microspectrometers, IEEE Trans. on Instr. and Meas. 53 (2004), pp. 197-202.

[15] R. Siebert, J. Müller, Infrared integrated optical evanescent field sensor for gas analysis Part I: System design, Sens. Actuators B 119 (2005), pp.137-148.

[16] G. de Graaf, W. der Vlist, R.F. Wolffenbuttel, Design and fabrication steps for a MEMS-based infrared spectrometer using evanescent wave sensing, Sens. Actuators A 142 (2008), pp. 211-216.

[17] C. Maims, J. Hulme, P.R. Fielden, N.J. Goddard, Grating coupled leaky waveguide mirco channel sensor chips for optical analysis, Sens. and Actuators B 77 (2001), pp. 971-978.

[18] W.A. Challener, J.D. Edwards, R.W. McGowan, J. Skorjanec, Z. Yang, A multilayer grating-based evanescent wave sensing technique, Sens. and Actuators B 71 (2000), pp.42-46.

[19] A. Agoston, C. Ötsch, J. Zhuravleva, B. Jakoby, An IR-Absorption Sensor System for the Determination of Engine, Proceedings IEEE Sensor 2004, pp.463-466.

[20] J. Kasberger, P. Rauter, T. Fromherz, B. Jakoby, A Slab Waveguide Sensor For a Fully Integrated IR-Absorption Sensor System – Theoretical and Experimental Verification, Proc. Transducers (2009), pp.999-1002.

[21] J. Kasberger, B. Jakoby, Design of a Novel Fully Integrated IR-Absorption Sensor System, Proc. IEEE Sensors (2007), pp.515-518.

[22] J. Kasberger, B. Jakoby, Grating-Coupling of Thermal Radiation as an Essential Element of a Fully Integrated.

[23] J. Kasberger, T. Fromherz, A. Saeed, B. Jakoby, Miniaturized Integrated Evanescent Field IR-Absorption Sensor: Design and Experimental Verification with Deteriorated Lubrication Oil, Vib.Spectrosc. (2011), doi:10.1016/j.vibspec.2011.01.003.

[24] J. Kasberger, A. Saeed, T. Fromherz, S. Katletz, B. Jakoby, Spectral Resolution of the Grating-Coupler of a Miniaturized Integrated Evanescent Field IR-Absorption Sensor, J. Quantum Electronics, accepted, 2011.