

MEMS-based miniature near-infrared spectrometer for application in environmental and food monitoring

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Abstract— We present a hybrid MEMS (micro-electromechanical systems) based spectrometer of the size of a sugar cube, fabricated at the clean room facilities of Fraunhofer IPMS, Dresden. This SGS (scanning grating spectrometer) is designed for integrated spectroscopic applications in the field of food quality analysis and food processing technology. Using another, larger, MEMS-based scanning grating spectrometer, we perform a series of test measurements of the absorption of different types of oil. Thereby, we demonstrate the suitability of this larger MEMS-based SGS for food quality analysis and establish a set of reference measurements. The optical parameters of the sugar cube-sized SGS are then evaluated against the requirements established in the above reference measurements. The spectral resolution of this device is still not sufficient for application in the near infrared, owing to the inaccuracy of one particular production step. Once these changes are in place, we are confident to achieve resolutions below 20 nm. In conclusion, we propose the miniaturised SGS as a mobile spectrometer for in-situ analysis integrated with a data processing system.

Keywords - scanning grating spectrometer, near infrared spectroscopy, miniaturization, hybrid integration, MEMS, integrated optics, food analysis, materials analysis, environmental monitoring

INTRODUCTION

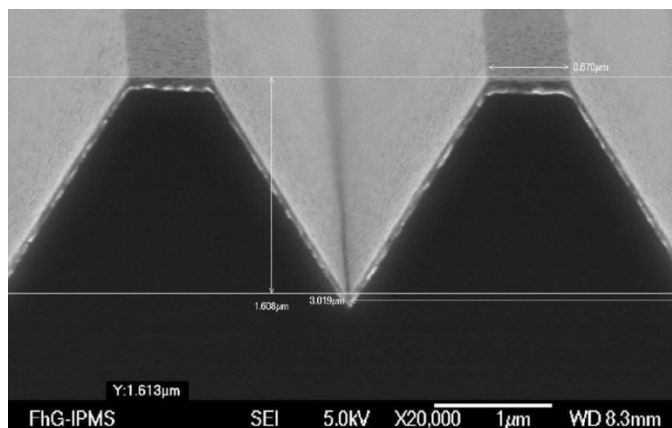
Near-infrared (NIR) spectroscopy is ubiquitous in the value chains supplying everyday life, with applications in food and environmental monitoring, medical care and industrial measurement technology. The technique facilitates non-destructive testing, and a qualitative as well as quantitative analysis of a large variety of compounds. During the last decades, the growing demand for such applications, placed under harsh ambient conditions, in confined spaces or with a need for portability has set new challenges for the development of suitable NIR spectrometers. In addition, potential high-volume applications and the trend towards integrated optical devices call for new concepts of spectrometer fabrication. We address this demand with the manufacture of a MEMS-based scanning grating spectrometer, with an integrated InGaAs diode for detection in the near infrared spectrum.

Scanning grating spectrometers (SGS) are among the most frequently applied spectroscopic instruments, with numerous

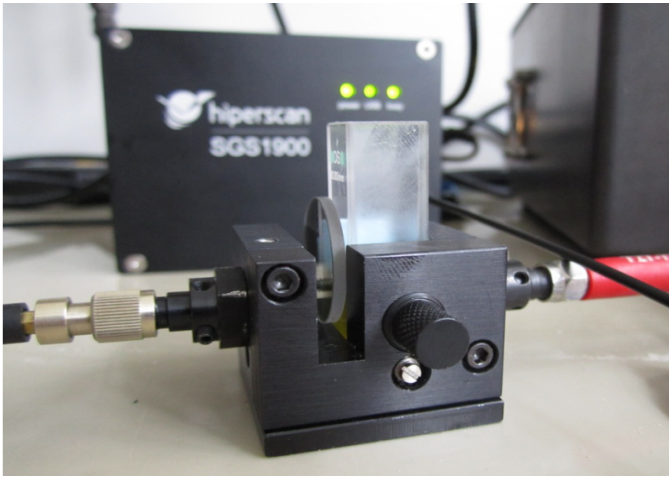
descriptions and advancements published [1,2]. Micro-electromechanical systems (MEMS) technology is capable of producing and assembling scanning gratings. Combining the two, a scanning grating spectrometer can be fabricated [3], which benefits from the energy and cost efficiency inherent to MEMS components. We explore the suitability of such a spectrometer for food and environmental monitoring. In particular, we perform measurements with a MEMS-based SGS on mixtures of different types of oil. Further areas of application include measurements of binary ethanol-water and glucose-water systems.

The above spectrometer is a modern, compact device exhibiting a volume of only a few hundred cubic centimetres and a few Watts of power consumption [4]. However, the intrinsic minimal size of a MEMS-based scanning grating opens the path to a further, extreme miniaturisation of a scanning grating spectrometer. Recently, we demonstrated such a miniaturised NIR spectrometer of the size of a sugar cube and with only a few milliwatts of power consumption [5]. This device fulfils both the optical design constraints of an SGS [6] and the need for production practicality during MEMS fabrication.

In a test measurement, the optical parameters of the miniaturised MEMS-based NIR spectrometer are evaluated for their use in food monitoring. An extremely miniaturised NIR spectrometer like this bears the potential for widespread use in



V-groove structures fabricated by anisotropic TMAH etching.



HiperScan SGS 1900 spectrometer and fibre-coupled cuvette holder.

food quality control or materials identification using integrated or handheld devices.

MEMS-BASED SCANNING GRATING SPECTROMETER

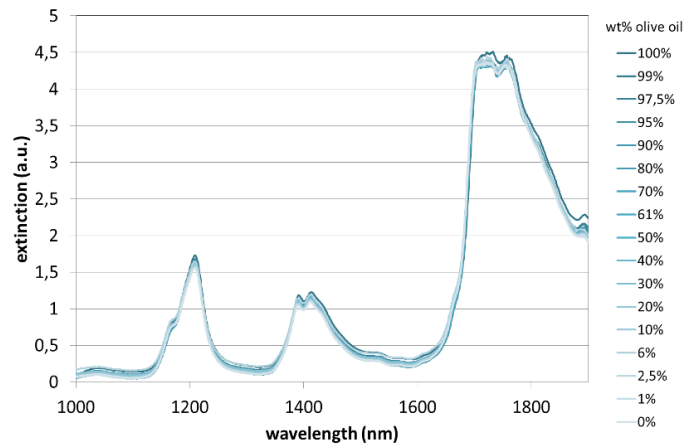
As a reference spectrometer for near-infrared absorption measurements, we use the HiperScan SGS 1900 scanning grating spectrometer. Its key component is a MEMS scanning grating, operating via a resonant electrostatic driving mechanism.

Fig. 1 shows a micrograph of a typical scanning grating fabricated at the clean room facilities at Fraunhofer IPMS. Equipped with an uncooled InGaAs photodiode, the spectrometer is designed for operation between 1000 and 1900 nm, with a spectral resolution of 10 nm. A fibre-coupled halogen lamp serves as the light source for absorption measurements. Its light is transmitted through a 10 mm optical glass cuvette and coupled into the spectrometer via optical fibre, as shown in Fig. 2.

Overall, this spectrometer is lightweight and compact, with a size of just (105 x 80 x 86) mm³. Owing to the MEMS component which replaces an expensive grating, the device is very cost efficient. Nevertheless, as we briefly demonstrate below, its sensitivity and resolution easily suffice for the tasks commonly addressed by NIR spectroscopy.

NIR ABSORPTION MEASUREMENTS OF OIL BLENDS

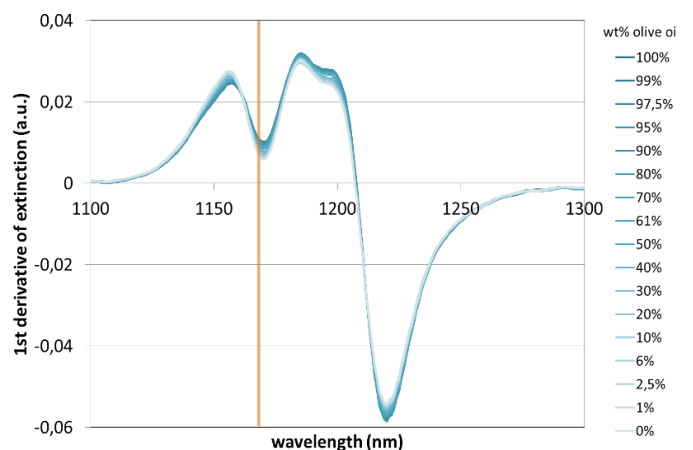
Food quality control is a field where NIR spectroscopy is widely applied, in particular for monitoring the composition of agricultural produce, which may be subject to seasonal variation or suffer from adulteration. Prominent examples are the detection of adulteration in beef [7] or the control of the ethanol content of beer [8]. As an integral constituent of the Mediterranean diet and associated with a healthy long life, olive oil is exported from the Mediterranean across the world. According to FAO (2004), olive is the most extensively cultivated fruit crop worldwide.



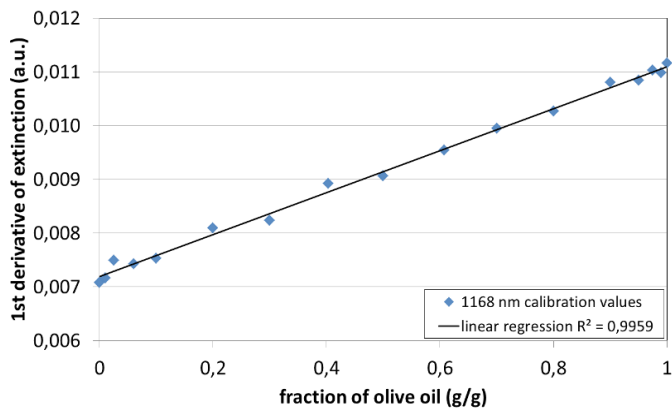
Extinction spectra of olive oil/rapeseed oil blends, ranging from 100% olive oil (dark) to 0% olive oil content (light), measured using the HiperScan SGS 1900 MEMS-based NIR spectrometer.

NIR spectroscopy is applied in the olive oil industry for quality control [9]. To test the practical use of a MEMS-based spectrometer, we chose a simple binary system of one type of olive oil and one type of rapeseed oil. Both substances were picked from a range of commercially available olive and rapeseed oils, respectively, for their representative NIR spectrum. The oils were mixed by weight into homogeneous blends, no phase separation occurred.

Fig. 3 shows the extinction spectra of the blended samples recorded from the transmission of a halogen source through a 10 mm cuvette. As shown by the consistently low background, the fraction of scattered light is small. However, as we did not attempt to monitor the scattered fraction, we must speak of extinction instead of absorption, even if the signal obtained mainly originates from NIR absorption. Characteristic absorption progressions are clearly evident around 1210, 1420 and 1730 nm. The bands at 1210 nm are associated with the 2nd overtone of the C-H stretching vibrations and those between 1700 and 1760 nm correspond to the first overtone of the C-H stretching mode of the methyl, methylene and ethylene moieties present [10]. Despite the rich spectral features, no significant differences are apparent to the naked eye, when



First derivative of the extinction spectra shown in Fig. 3. The orange line indicates the spectral position used for the binary model.

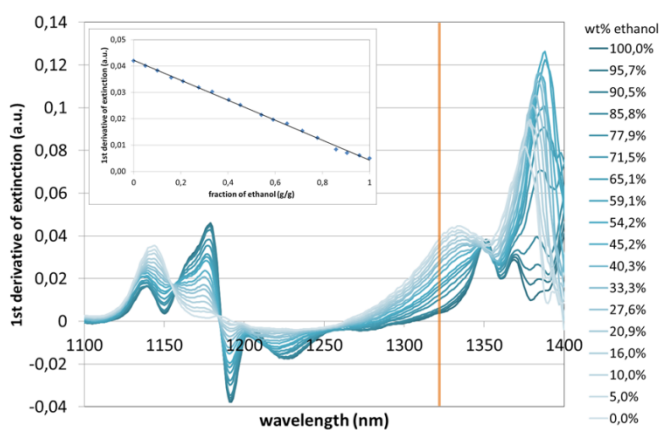


The values of the first derivative of the extinction spectra at 1168 nm show excellent correlation with the fraction of olive oil in the blend.

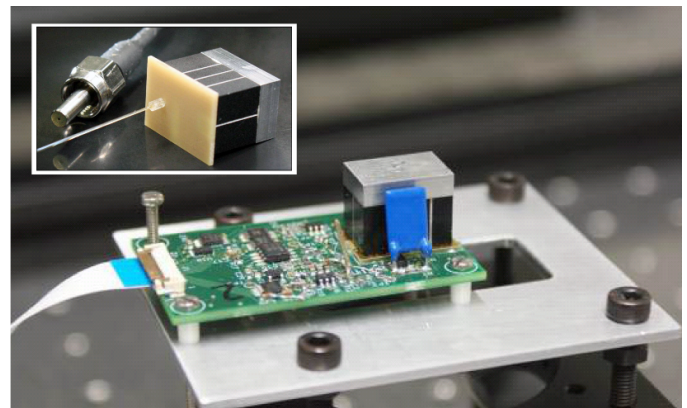
comparing the extinction spectra of the samples containing 100% olive oil (darkest curve) and 100% rapeseed oil (lightest curve).

However, by calculating the first derivative of the extinction spectra, we reveal a distinct trend followed by the peaks around 1210 nm, as shown in Fig. 5. A similar monotonous behaviour is observed in other spectral regions (not shown). For a first approximation of a binary model, we select the wavelength 1168 nm of the derivative spectra. Fig. 6 shows the resulting “calibration” of derivative amplitudes at 1168 nm to the concentration of olive oil in the sample with an excellent R^2 of 0.9959. The sensitivity of detecting olive in rapeseed oil and vice versa is estimated to be 3 % wt./wt.. Considering that this ad-hoc analysis is crude at best, we expect that a rigorous PCA analysis yields unambiguous evidence of the quality of the spectral measurements.

In order to cover a wider range of test substances, measurements analogous to the above were performed on binary mixtures of Ethanol and water. Fig. 6 illustrates that, again, the values of the derivative spectra correlate excellently with the Ethanol content of the mixtures. The same applies to



First derivative of NIR extinction spectra of binary mixtures of Ethanol and water. Inset: The values of the derivative spectra at 1327 nm correlate with the Ethanol content of the mixtures, $R^2=0,9983$.



Miniaturised optical bench of the spectrometer on drive and amplifier board. Inset: Unmounted optical bench of the hybrid-integrated MEMS spectrometer complete with fibre and SMA mount.

binary mixtures of Glucose and water, which is not shown here for brevity. Ethanol and Glucose are substances highly relevant in monitoring the composition of a wide range of foodstuffs. Using the SGS 1900 spectrometer and a very basic binary model, we are able to identify their concentration to an accuracy of 2 % wt./wt.

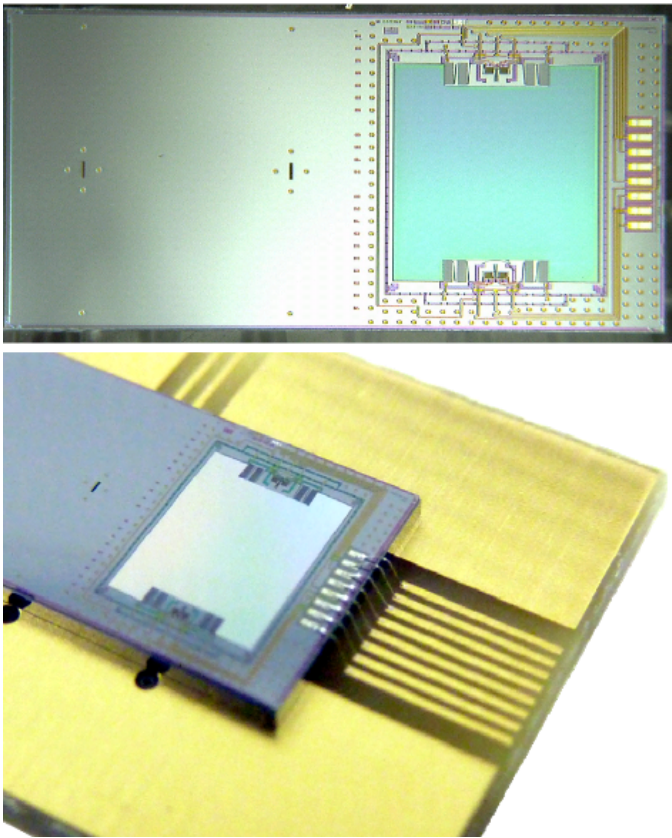
In conclusion, we find that a MEMS-based scanning grating spectrometer is capable of recording experimental data of a respectable quality.

MINIATURISED MEMS-BASED SPECTROMETER

For integrated optical applications, ultimate portability and mass produced spectrometers, it is highly desirable to achieve the functionality demonstrated above with an extremely downsized spectrometer. In principle, the typical size of MEMS gratings of up to few millimetres allows proceeding in this direction.

Such a miniaturised spectrometer was fabricated entirely at facilities of Fraunhofer IPMS, Dresden [11]. It is designed to be portable, integrable into handheld devices and battery powered. As in the instrument tested above, the key component of the miniature SGS is a MEMS scanning grating, which operates via a resonant electrostatic driving mechanism. The plate into which the grating is etched is 75 μm thin and designed to oscillate at 125 Hz. Its lateral dimensions are limited by considerations of mechanical stability, to few millimetres. Technological and optical aspects define the grating constant: It must be wider than $\lambda/2$ – we chose 1.6 μm , corresponding to approximately 700 lines / mm, which is feasible for the anisotropic etching process. In addition, this relatively large grating constant opens the possibility to use the full range of the InGaAs detector, if needed.

The development targets for the MEMS based spectroscopic system comprised a volume below 25 x 25 x 12.5 mm^3 (half a cubic inch) and a power consumption of a few milliwatts. In terms of spectral resolution, the NIR applications in mind require approximately 10 nm in the wavelength range of 1000 nm to 1900 nm. First order calculations were performed based on these external

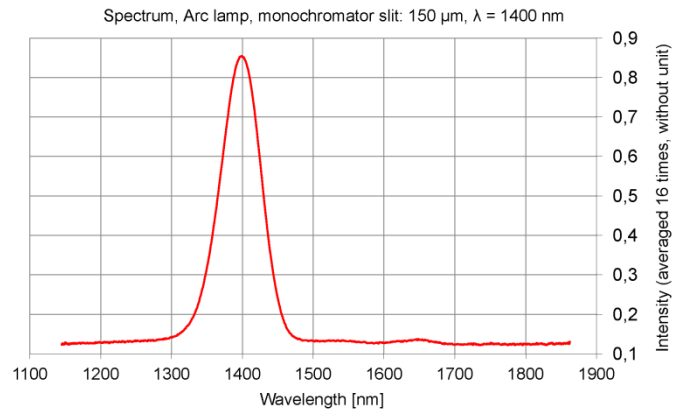


Top: MEMS scanning grating chip, with a size of (5.3 x 9.5) mm², from left to right: entrance slit, exit slit, grating plate with integrated position detection. The axis of grating rotation runs vertical through the grating. Bottom: MEMS-chip mounted on a PCB.

parameters, resulting in a focal length of 8 mm, as a suitable compromise between size, resolution and technological aspects.

In the actual design of the spectrometer configuration, these targets had to be implemented. Here, the task of miniaturising a MEMS-based system required a twofold approach: First, the MEMS chip itself had to be laid out optimally for a spectrometer design. As shown in Fig. 8 above, the functionality of the grating chip is completed by two optical slits and includes a piezoresistive position detection.

The second important issue is the feasibility to manufacture and assemble the spectrometer by using established MEMS processes, as this is the only way to ensure accurate reproducibility of all processing steps and remain cost efficient at the same time. Therefore, we chose a planar mounting approach, where the required substrates are stacked on top of each other [11]. The actual optical design of the spectrometer configuration was optimised using ZEMAX software. Here, the common W-configuration of a Czerny-Turner type spectrometer turned out to be impractical for use with a MEMS chip [5]. In all physically relevant configurations, such a setup sets the grating far off axis. As apparent from Fig. 8, the spatially fixed integrated design with slits and grating in the same plane (on the chip) does not allow for tilting the rest position of the grating. A further restriction is that the deflection of a MEMS grating is always symmetrical about the



Spectrum of the monochromated output of a short arc lamp, wavelength set to 1400 nm, slit width 150 μ m, detection using the hybrid-integrated MEMS spectrometer.

rest position. Therefore, the entrance and exit angles of the spectrometer must be adjusted via the mirrors. As a consequence, both must be positioned to the same side of the grating, causing an asymmetrical optical design [5].

CHARACTERISATION OF THE MINIATURE SPECTROMETER

The miniaturised MEMS spectrometer was characterised by detecting the monochromated output of a short arc lamp. The spectrum obtained is shown in Fig. 9. In summary, we obtain

- a spectral range from 950 to 1900 nm
- a spectral resolution between 54 and 65 nm, depending on the spectral position
- a grating deflection of $\pm 9.5^\circ$
- an outer instrument size of (17 x 12 x 16) mm³

Comparing these parameters to the performance of the HiperScan spectrometer tested above and to the development targets, the main shortcoming of the miniature spectrometer is its spectral resolution. Considering that the spectral width of the NIR absorption peaks shown in Fig. 3 is below 20 nm, a further reduction of the spectral resolution by a factor of 3 to 5 is desirable. This is somewhat unexpected as the optical design of the spectrometer was optimised for a resolution comparable to the SGS 1900.

We suggest that the final assembly step during spectrometer production is a main cause of the insufficient spectral resolution: This assembly step is not sufficiently accurate as it is currently monitored using a camera system with a limited focal depth on a device which exhibits a finite (12 mm) height. Therefore, an improvement of the assembling accuracy is expected to result in a much better spectral resolution, in line with the optical calculations. In particular, this will require an active control of the final spectrometer assembly.

CONCLUSIONS AND OUTLOOK

In conclusion, the miniaturized MEMS based scanning grating spectrometer is a portable, integrable, energy efficient device. It is suitable for a range of NIR applications that require a handheld device or must operate in a confined space, from medical services to food quality analysis or materials identification.

We designed, fabricated and successfully tested a prototype of a hybrid-integrated MEMS spectrometer. Unfortunately, the current design does not entirely fulfil the specifications set by standard NIR applications. In particular, the resolution must be further improved. In the future, we plan an active control of the final assembly step to enhance the reproducibility and accuracy of the production process.

REFERENCES

- C. Palmer, *Diffraction grating handbook*, 6th ed., Newport, 2005.
- M.A. Gil, and J.M. Simon, "New plane grating monochromator with off-axis parabolical mirrors", *Applied Optics*, vol. 22, 152-158, 1983.
- H. Gröger, A. Wolter, T. Schuster, H. Schenk, and H. Lakner, "Realization of a spectrometer with micromachined scanning grating", *Proc. of SPIE*, vol. 4945, 46-53, 2003.
- Data sheet of SGS 1900 spectrometer, www.hiperscan.com, 2014.
- H. Gröger, J. Knobbe, T. Pügner, and H. Schenk, "Design and characterisation of a hybrid-integrated MEMS scanning grating spectrometer", *Proc. of SPIE*, vol. 8616, 8616-0L, 2013.
- T. Pügner, J. Knobbe, and H. Lakner, "Basic angles in microelectromechanical system scanning grating spectrometers," *Applied Optics*, vol. 50, no.24, August 2011.
- J. McElhinney, G. Downey, and C. O'Donnell, "Quantitation of Lamb Content in Mixtures with Raw Minced Beef Using Visible, Near and Mid-Infrared Spectroscopy", *Journal of Food Science*, vol. 64, no. 4, 587-591, July 1999.
- S. Engelhard, H.G. Löhmansröben, and F. Schael, "Quantifying ethanol content of beer using interpretive near-infrared spectroscopy", *Applied Spectroscopy*, vol. 58, no. 10, 1205-1209, October 2004.
- S. Armenta, J. Moros, S. Garrigues, and M. De La Guardia, "The use of near infrared spectroscopy in the olive oil industry", *Critical Reviews in Food Science and Nutrition*, vol. 50, no. 6, 567-582, 2010.
- G. Downey, P. McIntyre, and A.N. Davies, "Detecting and Quantifying Sunflower Oil Adulteration in Extra Virgin Olive Oils from the Eastern Mediterranean by Visible and Near-Infrared Spectroscopy", *Journal of Agricultural and Food Chemistry*, vol. 50, no. 20, 5520-5525, 2002.
- T.Pügner, J. Knobbe, H. Gröger, and H. Schenk, "Realization of a hybrid-integrated MEMS scanning grating spectrometer", *Proc. of SPIE* vol. 8374, 83740W-1, 2012.