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Fiber-Coupled MEMS-based NIR Spectrometers for Material Characterization in Industrial Environments

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Abstract Recently emerged near-infrared (NIR) spectrometers based on micro-electromechanical systems (MEMS) are a highly compact, rugged and cost-efficient alternative to infrared spectrometers conventionally used in industrial environments. The majority of the devices currently available are designed for measurements in diffuse reflection geometry in close contact with the sample, with built-in low-power halogen light sources – usually intended for consumer applications. However, for most material characterization applications in the industrial environment such a measurement configuration is neither feasible, nor practical. Using light transmitting optical fibers in combination with a measurement probe and a high power fiber-coupled light source is often preferable. In this contribution we compare various fiber-coupled NIR-spectrometers based on different MEMS technologies and demonstrate the applicability of MEMS spectrometer technology in industrial environments.

Keywords NIR Spectroscopy, MEMS Technology, inline monitoring, fiber-coupled, partial least squares regression

1 Introduction

Near infrared (NIR) spectroscopy is one of the most frequently used tools in industry to gain real-time information about the chemical or physical state during production processes. It allows for accurate monitoring and tight process control to achieve maximum process efficiency and product quality. Besides its inline-capability, non-destructive nature and low running costs, it allows to measure multiple process parameters simultaneously through the use of multivariate data analysis techniques [1]. One major obstacle for the implementation of NIR spectroscopy remains the relatively high hardware costs of conventionally used process spectrometers. Modern MEMS-based NIR spectrometers offer a rugged and compact alternative to conventional process spectrometers at a fraction of the price (about 1/10th) without any moving parts, which is an additional advantage in industrial environments. This relatively new technology has already proven capable of replacing conventional spectrometers in certain demanding applications [2–4]. However, most of the MEMS-based NIR spectrometers available today are designed for measurements in reflection geometry using built-in low power halogen light sources. Performance comparisons for the different types of these spectrometers can be found in the literature [5]. While this measurement geometry is suitable for solid samples that can be directly accessed e.g. in handheld measurements, it is often not suitable or practical for industrial applications. Often high power light sources in combination with inline measurement probes connected via light transmitting fibers are necessary or much more convenient.

In this contribution different fiber-coupled MEMS-based spectrometers are compared in terms of covered wavelength range, metrological properties and overall performance. Furthermore, the applicability of MEMS-based spectrometers in an industrial setting is demonstrated. This should shed some light on the upsides and downsides of the different available technologies and measurement principles when used in a fiber-coupled configuration.

2 Experimental

Seven individual ultra-compact fiber-based NIR-spectrometers that use three different MEMS-based technologies for spectral light acquisition were tested. Two spectrometers rely on digital light processing (DLP), one on Fourier transform infrared (FTIR) spectroscopy and four on tunable Fabry-Pérot (FP) filter technology. All spectrometers were simply connected via USB to a PC and require no additional power source. For the comparison of these different MEMS-spectrometers, a simple setup was built consisting of a fiber-coupled halogen light source, a sample holder and two low-OH SMA-soupled fibers for connecting the light source and MEMS-spectrometer, respectively. A schematic drawing of the experimental setup is shown in Fig.2.1.

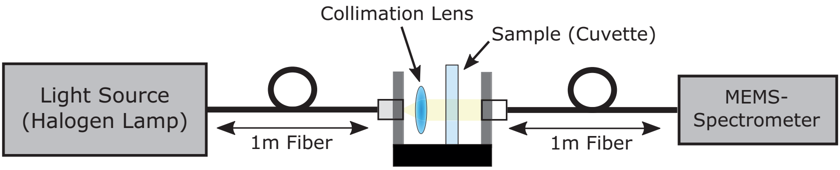

Figure 2.1: Schematic drawing of the measurement setup used to compare the different MEMS-based spectrometers. For details see text.

The fiber on the left was kept the same for all measurements (low-OH, 600µm core diameter). The same fiber was used on the right for the MEMS-FTIR and the FP spectrometers, but was exchanged for a 7-to-1 round to linear fiber for the DLP spectrometers (low-OH 200µm core diameter for each fiber). This was done to ensure maximum light coupling into the grating-based DLP-spectrometers which have an entrance slit that be illuminated much more effectively using a linear arrangement of fibers on the spectrometer end. For the measurement of the 100%-lines (Fig.3.1), no sample was inserted and the light of the fiber-coupled light source was measured directly, while for the sample spectrum shown in Fig.3.2, ethanol was put into a Infrasil quartz cuvette with a light path inside the sample of 1mm. Measurement settings for each spectrometer were individually set to get an acquisition time of 2 seconds for each spectrum.

3 Results and Discussion

The setup shown in Fig. 2.1 without any sample cuvette was used to acquire 101 individual spectra for each spectrometer. These spectra were used to calculate 100%-lines $A^{100\%}$, where the first measured spectrum

S_0 is used as background which results in 100 lines $A_i^{100\%}$ given by:

$$A_i^{100\%} = \log_{10}(S_0) - \log_{10}(S_i) \tag{3.1}$$

The resulting 100%-lines for all tested spectrometers are plotted in Fig.3.1. Therein also the root mean square (RMS) of the 100%-lines for each of the spectrometers is indicated to allow for a better performance comparison. The RMS is obtained by averaging all the squared entries for all 100 obtained lines $A^{100\%}$:

$$\mathrm{RMS} = \frac{1}{100}\sum_{i=1}^{100}\left[\frac{1}{N_\lambda}\sum_{j=1}^{N_\lambda}\left[A_i^{100\%}(\lambda_j)\right]^2\right]^{1/2} \tag{3.2}$$

where N_λ is the total number of spectral points acquired with the respective spectrometer.

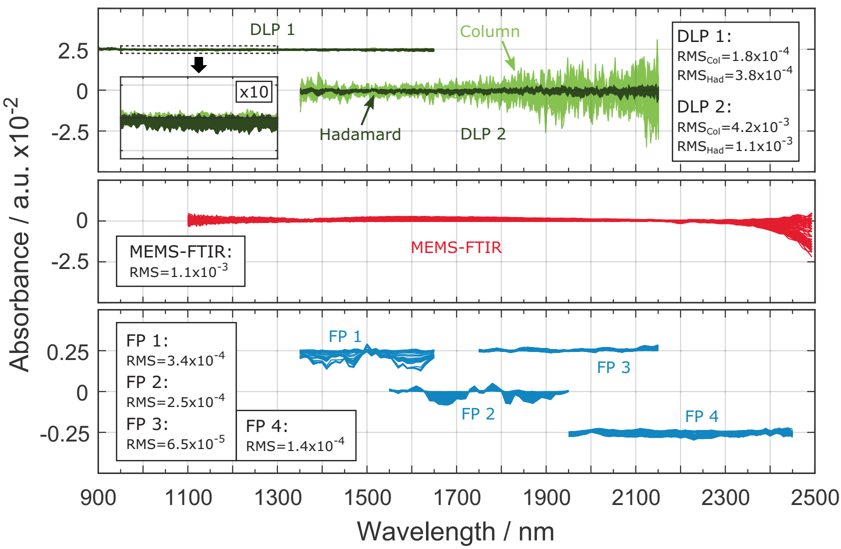

Figure 3.1: 100%-lines recorded with the different MEMS NIR spectrometers. The data for some of the spectrometers have been vertically shifted (DLP 1, FP1, FP 3, FP 4) for better visibility. RMS values for the spectrometers are given to allow for easier performance comparison.

The uppermost graph in Fig.3.1 shows the 100%-lines for the two investigated DLP-spectrometers. The first one (DLP 1) covers the wavelength region from 900nm-1650nm which is a very common region for NIR-spectrometers since it is the spectral response range of typical InGaAs detectors. The second DLP device (DLP 2) covers the spectral region from 1350nm-2150nm which requires an extended InGaAs detector. Also the sizes of both devices differ, with DLP 2 having a size of only 59.5x47.5x24.5mm^3 while DLP 1 has the dimensions 75x58x26.5mm^3 (≈66% bigger). Due to the measurement principle used in both of these devices, different measurement modes can be used, namely the *Column* mode and *Hadamard* mode. The latter has a multiplex advantage resulting from collecting light from 50% of all available wavelengths at a time using Hadamard patterns on the digital mirror device (DMD) instead of single columns that project only a small wavelength band onto the photodetector. Both measurement modes were tested using the same measurement parameters (resolution, exposure time etc.) and are shown in Fig.3.1 in bright green (Column) and dark green (Hadamard), respectively. It can be seen that the shift to longer wavelengths and the more compact form factor does not come without trade-offs when it comes to noise performance. The RMS of the 100%-line is one or two orders of magnitude smaller for DLP 1 for Hadamard and Column mode, respectively. Interestingly, the multiplex advantage does not affect the performance of DLP 1, in fact the noise performance when measuring in Column mode is even better than in Hadamard mode, suggesting other noise sources that are not influenced by the multiplex advantage e.g. influence of temperature drifts. A clear demonstration of the benefits of multiplexing can be seen in the data for DLP 2, where the detector noise is the main noise source. Here the RMS is about four times better when using Hadamard mode instead of Column mode.

The middle graph visualizes the data obtained with the MEMS-FTIR device. This device covers the broadest spectral range of all tested spectrometers, namely 1100nm-2500nm using an extended InGaAs detector and has the largest size of 76x57x49mm^3. Also the FTIR measurement principle used in this device has multiplex advantage, which allows for good noise performance similar to the performance using the multiplexing Hadamard mode with the DLP 2 spectrometer. The larger noise level at wavelengths above 2300nm can be partially explained by the low light intensity in this wavelength range. Not only does the halogen light source (2850K color temperature) emit most of its intensity at shorter wavelengths, but also the used low-OH fibers show significant light absorption for wavelengths longer than 2300nm.

The third kind of tested MEMS-spectrometers relies on tunable FP-filters. This measurement principle results in the smallest wavelength coverage, which is why four individual spectrometer modules are required to cover the wavelength range from 1350nm-2450nm. An advantage of this technology is that it can be realized in an extremely compact housing ($25x25x25mm^3$) and also has the lowest hardware costs. As visible in the bottom graph in Fig.3.1, another upside of this technology is the superior noise performance over the whole wavelength region. All tested FP-spectrometers have RMS values in on the order of 10^{-4} or lower similar to the performance of DLP 1, but also at longer wavelengths where commonly lower light intensities are encountered.

To get a better idea about how the measurement technology shapes acquired absorption spectra ethanol was used as a sample since it shows multiple absorption bands across the whole NIR spectral range. As background the measured spectrum without any sample was used. As a reference, the absorption spectra of ethanol measured with a Bruker Vertex 70 benchtop FTIR spectrometer using the same cuvette (without any fibers and using its internal light source) are shown in gray. The acquired data can be seen in Fig.3.2.

Spectra acquired with the individual MEMS-spectrometers overlap very nicely and show the same features at the same spectral positions. Furthermore good agreement with the reference spectrum measured using the benchtop FTIR was achieved. The spectra recorded with the FP-filter spectromters (blue) tend to show flattened spectral features due to the lower spectral resolution of approximately 15nm-30nm, compared to the MEMS-FTIR (5nm-13nm) and the DLP spectrometers (10nm-15nm).

The biggest differences between the recorded spectra can be seen in the wavelength region above 2200nm. Here the FP-based spectrometer strongly underestimates the strength of the absorption of ethanol. However, the main spectral features can still be seen albeit with significantly lower resolution, such as the shoulder at 2350nm resulting from the sharp absorption band at this wavelength visible in both the MEMS-FTIR and benchtop FTIR spectrum. The lack of multiplex ad-

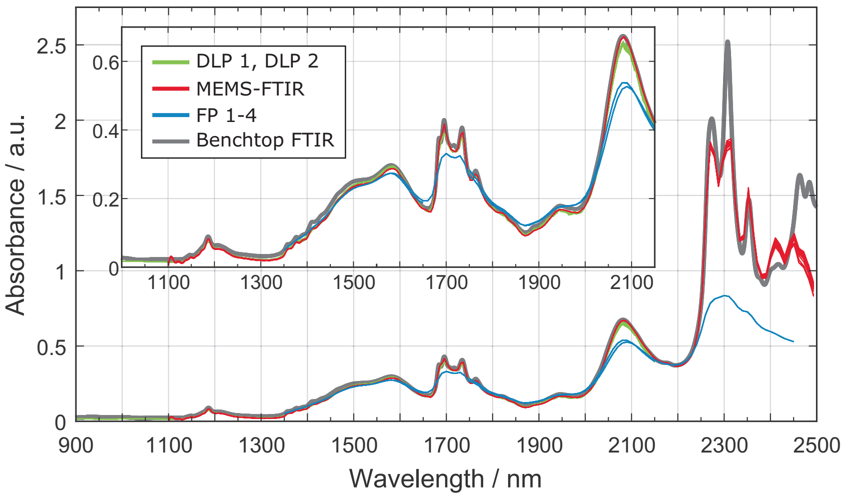

Figure 3.2: Absortption spectrum of ethanol recorded with the different MEMS NIR spectrometers (green, red, blue) and a benchtop FTIR (gray). The inset shows a zoom of the wavelength region 1000nm-2150nm.

vantage that comes with the FP-based measurement technique can also cause problems in this wavelength region. Since the light intensity is very low due to low emission from the light source, high absorption from the used fibers and additionally strong absorption from the ethanol sample, the measured signal is rather weak and non-linearities of the used detector or electronics can cause deviations from the real absorption spectrum.

Slight differences can also be seen between the MEMS-FTIR and benchtop FTIR caused by the lower resolution of the MEMS-FTIR which leads to the flattening of the sharp absorption bands around 2300nm. Furthermore, the much lower light intensity at long wavelengths due to the absorption in the used fibers (not present for the benchtop device) does not allow for a good measurement of the ethanol absorption bands above 2400nm. This problem could be solved e.g. by using more expensive zirconium fluoride (ZrF_4) fibers to reduce light attenuation.

4 Inline Process Monitoring using Fiber-Based MEMS-Spectrometer Technology

In this section measurement data from an industrial application of a FP filter based MEMS-spectrometer will be presented to demonstrate the potential of MEMS technology to replace commonly used FTIR-process spectrometers in the industrial environment. As a use-case, NIR-monitoring of a batch-wise melamine formaldehyde resin production plant was chosen. At Metadynea Austria GmbH, the resin production is routinely monitored and controlled in real time using NIR spectroscopy in combination with partial least squares (PLS) regression [6]. The combination of NIR spectroscopy and PLS regression is e.g. used to determine the batch end point. This leads to a significant reduction of necessary offline measurements and helps to avoid out of spec batches.

To demonstrate the usability of FP-filter based MEMS spectrometer technology for the determination of the batch endpoint, the optical fiber was simply switched from the routinely used FTIR spectrometer to a suitable MEMS device and a PLS regression model was calibrated and validated using the data acquired with the FP spectrometer. The data is summarized in Fig.4.1. As can be seen from the data, the measurement quality is very similar for the two different spectrometers. The root mean squared error of prediction (RMSEP), which is an important parameter to judge the measurement performance, is higher for the MEMS spectrometer (0.044) when compared to the FTIR (0.024), but still clearly below the threshold value (0.1) set by the company. This means the low-cost MEMS-based spectrometer can be used to replace the conventional, much larger FTIR spectrometer while still delivering satisfying results.

5 Conclusion

In this contribution, seven individual fiber-coupled MEMS-based NIR spectrometers were compared in terms of their covered wavelength range, resolution and noise performance. The tested MEMS-spectrometers based on different measurement technologies, namely DLP, FTIR and tunable FP-filters. Each technology has its advantages

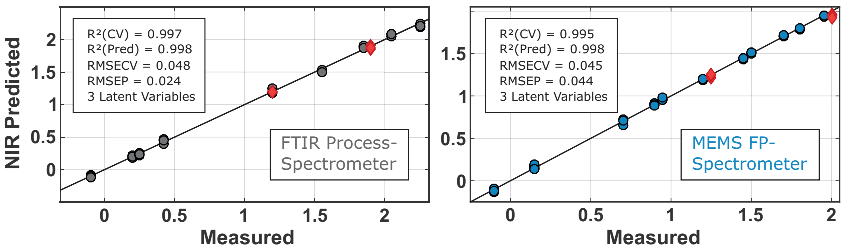

Figure 4.1: Data comparison between a conventional FTIR process spectrometer (left) and a low-cost FP spectrometer (right). Normalized values calculated from the PLS regression models are plotted versus offline measurements. Calibration data is shown in gray (FTIR) and blue (FP), respectively. Validation data is shown in red.

and disadvantages. While MEMS-spectromters based on tunable FP-filters tend to have the best noise performance, they also have lower spectral resolution and spectral coverage (300nm-500nm) and lack the multiplex advantage that can be exploited with DLP and FTIR technology. The broadest spectral coverage can be achieved using the FTIR measurement technique, while still maintaining very reasonable noise performance, but at higher hardware costs (about 3x). MEMS-spectrometers based on DLP-technology offer a good alternative by offering good spectral coverage of about 800nm while having comparable noise performance to the MEMS-FTIR when the multiplex advantage is exploited by measuring in Hadamard mode. When the right instrument is chosen for the specific application, fiber-based NIR MEMS-spectrometers show huge potential to allow for much more cost effective spectroscopic measurement solutions in the industrial environment without significant sacrifices in measurement performance. For this the presented application for inline batch monitoring at a resin production facility is a good example. There it was successfully shown that the narrow band FP devices were a suitable substitute for the conventional broadband FTIR spectrometer.

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