

Quality Monitoring of Diesel Exhaust Fluid in Vehicles using Diffractive Interference Sensors

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Abstract— In this paper, we describe a simple design for an optical sensing device based on differential interferometry that can be deployed as an in-line sensor for monitoring the quality of diesel exhaust fluid inside engines of diesel vehicles. This sensor can precisely determine (to within $\pm 1\%$ and lower) the percentage of urea in diesel exhaust fluid (DEF), which is a critical reactant in the selective catalytic reduction (SCR) process for reducing harmful nitrous oxide emissions from diesel vehicles into the environment. The operating principle is based on diffraction of laser light from a regularly spaced microarray. Preliminary performance results indicate that our sensor can precisely determine the concentration of urea in the DEF to within $\pm 0.0045\%$.

I. INTRODUCTION

The control and reduction of various pollutants that are released into the environment by burning fossil fuels is a top priority for governments around the world and diesel tailpipe emission standards have been established for the same. Selective catalytic reduction (SCR) is an after-treatment method for converting detrimental nitrous oxides (NO_x) in diesel exhaust emissions into nitrogen (which is harmless) and water [1]. Diesel Exhaust Fluid (DEF), which is also known by its registered trademark AdBlue, is a colorless, nonflammable aqueous solution of urea (32.5% urea and 67.5% deionized water) and is a required reactant in the SCR process inside the engines of diesel vehicles. The efficiency of the SCR process depends critically on the quality of the DEF, which needs to be injected into the exhaust stream downstream of the engine. There have been instances of adulterants (such as agricultural-grade urea or salt water) present in DEF as also situations where DEF has been unintentionally contaminated via inadequate storage methods (since DEF is corrosive for many metals and hence, needs to be stored carefully). The presence of unauthorized liquids results in low-quality DEF, or DEF that does not have the appropriate concentration of urea, and can lead to a complete breakdown of the SCR process via the formation of salts on the ceramic catalyst head. This results in the engine exceeding permissible emission levels leading to fines and penalties for the vehicle operator.

Other recently reported DEF quality evaluation approaches include techniques based on ultrasound (by measuring acoustic velocity propagation within a spatial volume of the solution) and electrical measurements (ac as well as dc measurements that are indicative of the ions that may be present in the solution as impurities) [2,3]. The operating principle of our sensing device, referred here as Diffractive Interference Sensing (DIS), is the differential response of the various diffracted orders to a refractive index (RI) change in the medium surrounding a micro-fabricated 2D diffraction grating.

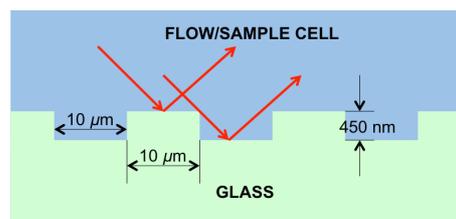


Fig. 1. Schematic representation of differential interference sensing element.

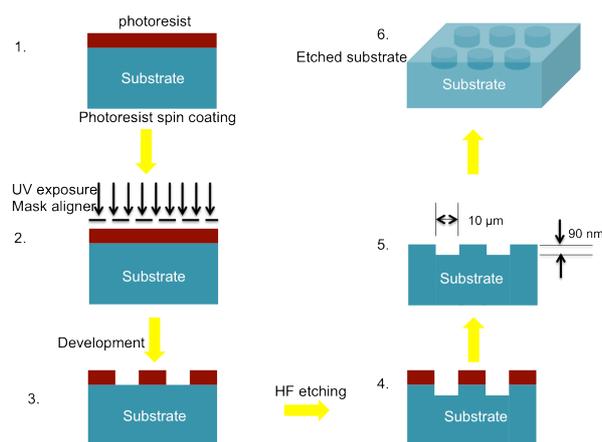


Fig. 2. Steps involved in the photolithographic fabrication procedure for the sensing element.

II. EXPERIMENTAL SETUP

The main element of the differential interference sensing device is a 1 cm-by-1 cm, 350 micron-thick glass substrate with a periodically patterned surface, depicted in Fig. 1. The surface patterning, defined via a mask photolithographically, consists of a two dimensional regular array of circular wells, which are 10 microns in diameter, 450 nm deep and spaced 10 microns apart on a positive tone photoresist deposited over the substrate. The exposed photoresist layer is subsequently etched using dilute hydrofluoric acid and then stripped with acetone resulting in the final pattern comprising of equally spaced micro-diffractive elements as shown in Fig. 2. In order to form a sample cell, an acrylic chamber was constructed on top of the glass chip for holding the test sample. A syringe pump was connected to this chamber via Teflon tubes so that the DEF could be diluted by the addition of de-ionized (DI) water. In our experimental setup, collimated, TM-polarized light from a helium-neon laser (wavelength: 633 nm; power: 5 mW) was intensity modulated with a chopper at a frequency of 1.5 kHz. This modulated laser beam was passed through the sample/flow cell to illuminate the patterned surface of the glass chip resulting in a diffraction pattern. The peak intensities of the zeroth and the first orders (I_0 and I_1 respectively) were roughly balanced using neutral density filters (3 and 0.6 OD respectively) and recorded on two identical Si photodetectors (Thorlabs DET100A), whose outputs were sent to two lock-in amplifiers (SRS830, Stanford Research Systems).

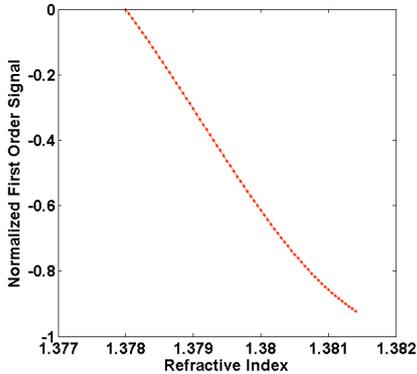


Fig. 3. Simulation of the expected signal change due to a 1% change in DEF urea concentration.

The fluid layer in contact with the grating surface fill up the wells on the sensor surface essentially creating a periodic variation of the reflection or transmission coefficient, $r(x,y)$ or $t(x,y)$ respectively, resulting in a diffraction pattern when probed with a laser beam in reflection or transmission mode. A change in the RI of the sample above the grating changes the periodic reflection or transmission coefficient, perturbing the diffraction pattern. As described previously [4], the zero order does not change significantly due to the change in sample RI. However, the first order signal being sensitive to the phase difference due to the grating depth responds to the change in RI and can be used to sense the sample RI.

Moreover, the zero order provides a stable baseline to correct for common mode noises and drifts arising from thermal and fluidic fluctuations in the sample cell [4]. The periodic $r(x,y)$ or $t(x,y)$ were calculated using a multilayer transfer matrix method and diffracted intensities were then calculated using Fraunhofer approximation [5]. Fig. 3, shows the result of this calculation for a sensor for the measurement of DEF urea concentration changes over 1% from the specified values. DEF is a solution of high-purity urea and DI water wherein the urea concentration is $32.5\% \pm 0.7\%$ with the RI range from about 1.381 to about 1.378 as measured with a benchtop refractometer. In other words, a 1% change over the specified values of urea concentration requires the measurement of refractive index to the 3rd decimal. Fig. 2 shows that a corresponding change in the fluid index surrounding the 2D grating will produce a signal change of nearly 90% from the baseline, which is well above the noise floor for measurement. Since the light passes through the (liquid) sample, any turbulence or inherent thermal and concentration gradients within the sample cell cause signal drifts that can overwhelm the effects from any RI variations. However, as the zero and first order diffracted beams have the same optical path, they serve as a mutual reference and by computing the ratio I_1/I_0 , such fluctuations due to systemic drifts can be eliminated.

Fig. 4 (a) depicts the change in the measured intensities as the composition of the sample within the flow cell is changed. The DEF solution in the sample cell was diluted with DI water in regular intervals such that the concentration of urea in the DEF decreased by 1% with each dilution step. As seen in Fig. 4 a), the ratio signal I_1/I_0 response follows in a step-wise manner as the concentration of the DEF is varied with dilution. Based on the signal-to noise ratio of the data shown in Fig. 4 a), we estimated a limit of detection (LoD), i.e. the smallest concentration change in the DEF solution, of about 0.0045%. Finally, we cross verified the data from DIS sensor with a commercial benchtop refractometer (Abbemat 200). Fig. 4 (b) shows the correlation between DIS signal plotted on the vertical axis and the refractometer values plotted on the horizontal axis. As we can see from Fig. 4 b) there is excellent correlation with the data from the DIS sensor and the refractometer indicating the applicability of DIS signals for high resolution refractometry. But more importantly, one can see the enhanced resolution of the DIS sensor relative to the benchtop refractometer from the significantly smaller vertical error bars compared to the horizontal error bars from the refractometer.

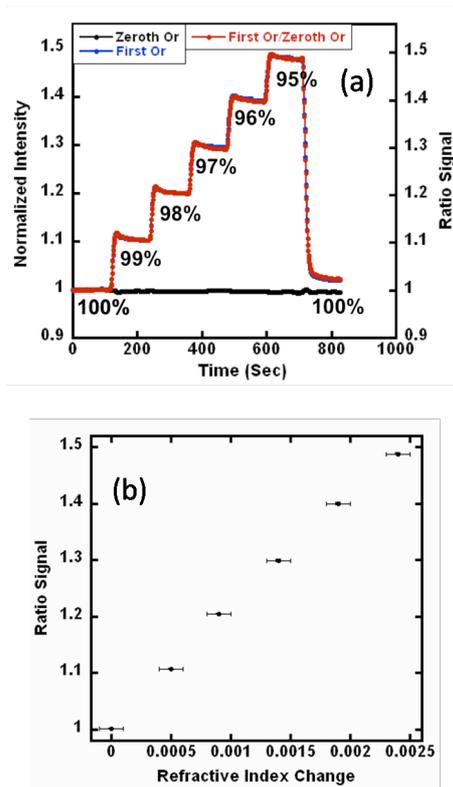


Fig. 4. (a) Plot of the I_1/I_0 ratio as a function of time. Each ‘step’ occurs when the DEF concentration is changed by 1% with DI water dilution. (b) Correlation of signal from the DIS sensor (errors indicated by horizontal bars) and the Abbemat refractometer (errors indicated by horizontal bars).

III. CONCLUSION

We present a simple, robust design for real-time quality sensing in DEF tanks inside diesel vehicles to meet legislated mandates for cleaner emissions. We detected changes in the urea concentration to within $\pm 1\%$ (which is typically the specification for the automotive industry to evaluate the

quality of DEF), which was well within our device sensitivity. Our device LoD corresponds to a 0.0045% change in the urea concentration, i.e., this value is the lowest concentration that we can measure. This technique can be extended for a similar analysis for several other fluids. Current DEF tanks come equipped with temperature and level sensors. Our next step would be to miniaturize and package the device as an in-line sensor to be placed either within storage containers or inside the tanks within the vehicle. We are developing ways to interface the sensor with a suitable data processing arrangement that can be integrated with some form of on board diagnostics system to indicate when the DEF in the tank needs to be replaced.

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REFERENCES

- [1] Clean Air Technology Center (MD-12) “Nitrogen Oxides (NOx), Why and How They Are Controlled,” Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency, EPA-456/F-99-006R, Nov. 1999.
- [2] *QLS RC Series Adblue/DEF Intank level and quality sensor*. Available: <http://www.meas-spec.com>
- [3] *Temperature ultrasonic level and concentration sensor for urea, DEF, and Adblue tanks*. Available: <http://www.ssitechnologies.com>
- [4] N. Kumawat, and M. M. Varma, “Differential Reflectance Modulation Sensing with Diffractive Microstructures,” *Appl. Physics Lett.*, vol. 101, 191103, 2012.
- [5] Max Born, and Emil Wolf, *Principles of Optics: Electromagnetic theory of propagation, interference and diffraction of light* (7th Ed.). Cambridge, UK: Cambridge University Press, 1999.