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Single nano-particle flow detection and velocimetry using Optical Feedback Interferometry

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Abstract—We present a sensing technique based on the Optical Feedback Interferometry (OFI) scheme in a laser diode that enables single particle detection at micro and nano-scales through the Doppler-Fizeau effect. Thanks to the proposed signal processing, this sensing technique can detect the presence of single spherical micro/nanoparticles and measure their velocity, even while their diameter is below half the laser wavelength. The method was validated with polystyrene spheres with diameter ranging from 196 nm to 10.14 μm flowing in diluted aqueous solutions. These results indicate potential applications for the biomedical and chemical engineering fields.

Keywords—Optical Feedback Interferometry, microfluidics, velocimetry, light scattering, nanoscale sensing, Doppler effect.

I. INTRODUCTION

The increasing demand for technologies of nanoparticles detection and characterization in biomedical and chemical engineering applications has reached industrial processes where production quality controls of materials and products requires dedicated tools. We propose here to apply a novel interferometric method to address this problematic.

The Optical Feedback Interferometry (OFI) in a laser diode arises when part of the emitted light is back-scattered from a distant object and then reinjected into the laser cavity. This reinjected light provokes a modulation of the laser output power that carries information about the light interaction with the target. This technique scheme has been earlier proposed in many metrology applications, such as displacement [1], velocity [2] and vibration [3]. Many efforts have been made in both experimental and theoretical frame in order to extend the application domains of such a sensor [4].

In the microfluidics field, many OFI sensing techniques are capable to perform flowmetry and local velocimetry [5]–[13]; but, to the best of our knowledge, none of them performed detection in a single particle, and especially with nanoparticles. Compared to other interferometry sensing schemes, the OFI offers great advantages of compactness, self-alignment and low-cost, and less and more simple external optical elements; especially for laser diodes with built-in package photodiode. While previous works on OFI flowmetry have been limited to high particle densities fluids in single or multiple scattering regimes, the work depicted in this paper propose to investigate the feasibility for the detection and the measurement of a single nanoparticle velocity.

II. PRINCIPLE OF OFI SENSING IN SINGLE PARTICLE REGIME

A particle detection OFI sensor is basically a laser diode with focusing optics allowing for a sharp focus at the center of the microchannel region where a laminar flow is established as illustrated by the Fig. 1.

Fig. 1. OFI sensing scheme for single particle detection in a microchannel.

In OFI sensing for microfluidics applications at single particle regime, once a particle crosses the laser sensing volume with a velocity V_T , it generates a Doppler shift frequency (f_D) of backscattered reinjected light by

$$f_D = \frac{2V_T \cos \theta}{\lambda}, \quad (1)$$

where λ is the laser wavelength, and θ is the angle between the laser beam and the flow direction.

The dynamics of backscattering is ruled by the scattering cross-section of the particles against the laser power density of illumination, and their optical properties in the medium based on the Mie theory.

The particles are carried in a liquid pumped at constant flow rate into the channel. The particle concentration in such fluid is controlled to achieve a single particle regime which requires that just one single particle is crossing the sensing volume during each signal acquisition period. The velocity V_T of each particle is dependent on its radial position in the duct where a laminar flow profile is established.

III. EXPERIMENTAL SETUP

The setup of OFI sensing used for single particle detection is represented in the Fig. 2. It is constituted by a 785 nm Fabry-Perot laser diode (Thorlabs L785P090) with a monitoring photodiode built-in package, custom made laser driver and

transimpedance amplifier and, a focusing lens (Thorlabs C240TME-B, 8 mm focal length) located at twice its focal distance regarding both the laser and the axial center of the micro-channel.

Fig. 2. OFI setup for single particle detection with $\theta=82^\circ$.

The microfluidic chip used in our experiments is a PDMS circular duct of 320 μm diameter, and 230 mm length. The related Reynolds number is about 2.64, which gives a hydrodynamic entrance region corresponding to 42.24 μm . The OFI sensor is set on a 3-axis linear stage (Zaber Tech. T-LSM100A), the laser beam was focused in the center of the micro-channel and far beyond the entrance region. In that way, the sensing volume is set over the region where the laminar velocity profile is fully developed fitting well the *Hagen-Poiseuille* profile [14] for a Newtonian fluid. For a circular cross section the maximum velocity (v_{max}), corresponding to the center of the channel, is equal to $2Q/A$; where Q is the volumetric flow rate, and A is the cross section area. In all experiments, the liquid was pumped at constant flow rate of 40 $\mu\text{L}/\text{min}$ with a syringe pump (Harvard Apparatus Pico 11 Plus). The angle θ of 82° is set between optical axis and the flow direction ensuring a maximum Doppler shift of 5.9 kHz for particle in the center of the duct. Such theoretical value is obtained by (1) at v_{max} .

As the current of the monitoring built-in package photodiode reveals an image of the output optical power of the laser, such current is acquired by a data acquisition card (National Instruments NI-6361 USB) through the custom transimpedance amplifier.

The signal processing is written with Matlab scripts embedded in a Labview VI. The whole detection algorithm is implemented in this environment. The graphical user interface (GUI) allows to configure the detection parameters, and to monitor the process in real-time.

The detection of the Doppler signal burst is based on a threshold condition on the signal amplitude in the frequency domain. Thus it consist in a band-pass filtering, a time-frequency conversion using Matlab Pwelch algorithm (window of 2^{13} samples, no overlapping, and 2^{17} DFT points), and a thresholding that keeps signal which amplitude is at least 3 dB above the noise floor in the spectrum. The band-pass filter cut-off frequency are set to 1 kHz and 8.5 kHz. The low frequency cut-off allows eliminating low-frequency perturbations. The high frequency cut-off is set based on the maximum Doppler shift expected with a 50% margin.

IV. EXPERIMENTS AND RESULTS

For the experiments, four different aqueous dispersions of monodispersed polystyrene (TPD=1.05g/cm³) spheres were tested. These Microparticles GmbH tracers are at unique controlled size with very low standard deviation (SD). The four diameters tested are: 196 nm (PS-R-KM10, 5% weight/volume, SD=5 nm), 552 nm (PS-R-L2654, 5% w/v, SD=13 nm), 4.89 μm (PS/Q-R-KM153, 10% w/v, SD=80 nm) and 10.14 μm (PS/Q-R-SC08, 10% w/v, SD=120 nm).

From each mother solution, we prepared a solution with a particle concentration low enough to reach a single particle regime regarding the time of acquisition: respectively 0.00005% w/v for 196 nm and 552 nm, and 0.01% w/v for the 4.92 μm and 10.14 μm . The first two sample solutions were diluted with deionized water, and the micrometric ones with

Fig. 3. OFI signals acquired with a unique PDMS particle sensing. Left column, time domain signal before (blue) and after (green) band-pass filtering; right column: filtered signal spectrum (blue solid line) compared to the sensor signal spectrum in absence of particle (black dashed line).

a) & b) 196 nm particle, $f_D=6.2$ kHz; c) & d) 552 nm particle, $f_D=4.5$ kHz; e) & f) 4.89 μm particle, $f_D=5$ kHz; g) & h) 10.14 μm particle, $f_D=4.2$ kHz.

PBS (phosphate buffer solution 1.05g/cm^3 , by Sigma-Aldrich) filtered by $0.2\text{ }\mu\text{m}$ membrane syringe filters.

The Fig. 3 shows an example of single particle that was automatically detected for each size. In the time domain signals, the blue and green lines represent respectively the original OFI signal and the filtered signal. In the spectra of the filtered signals, the black dashed lines correspond to the filtered signal spectrum acquired in absence of particle. These give a floor reference to be compared with the respective filtered spectrum (in blue) where there is particle detected.

To double check the purity of the used deionized water and filtered PBS against the eventual presence of spurious particles, we performed an experiment for each while maintaining the same detection parameters, no false positive was detected over 4 hours.

V. CONCLUSION AND FUTURE WORKS

An innovating OFI sensing scheme for detection of single particle able to address nanoscale particles is presented in this work.

Regarding other classical technologies for particle detection, the OFI technique, due to the fact that it is an optical non-invasive method, presents the benefits of higher capability of integration and compactness.

Among the main potential applications of this innovating technique, we could mention: flow cytometry; real-time cell counting; velocimetry at chemical micro-reactors for production of chemical substances and pharmaceuticals; biosensing for sickness diagnosis; detection of solid contaminants in fluid flows, like for water quality control; and also for prospections at marine science researches.

Perspectives of this approach are related to the exploitation of the burst properties in order to eventually perform particle sizing.

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