STSM REPORT

STSM application number: COST-ONLINE_STSM-BM1205-16456

STSM Grantee: Francisco Javier Azcona Guerrero.

STSM title: An experiment to prove the DSMI method as an approach for fluid media characterization.

Home institution: Centre for Sensors, Instruments and Systems Development (CD6), UPC-BarcelonaTech

Host institution: École nationale supérieure d'électronique, d'électrotechnique, d'informatique, d'hydraulique et des télécommunications (ENSEEIHT).

STSM period: March 30th 2014 to April 11th 2014.

STSM purpose:

To prove differential self-mixing interferometry (DSMI) as a method to measure small changes of the refractive index (Δn) in fluid media. The difference on the refractive index can then be related to some of the physical properties of the analyzed media such as concentration, temperature, etc.

Description of the work carried out during the STSM:

The work was divided into two major subjects:

- *Theory and simulations:*

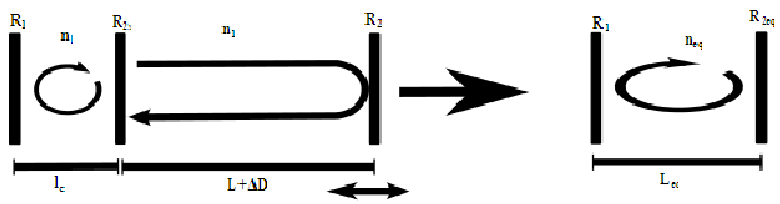

Fig. 1 Double cavity Fabry-Perot system used to model SMI.

In classical self-mixing interferometry (SMI), it is possible to model the interactions of the laser with the measured vibration as a double cavity Fabry-Perot system as the one shown in Fig. 1. Taking into account the requirements to produce lasing, it is possible to derive the excess phase (Eq. 1) and the resulting laser power (Eq.2) as:

$$0 = \Delta\varphi = 2\pi(\nu - \nu_0)\tau_e - C\sin[2\pi\nu\tau_e + \operatorname{atan}(\alpha)], \quad (1)$$

$$P_F = P_0[1 + m\cos(2\pi\nu\tau_e)], \quad (2)$$

where C accounts for the feedback factor, ν_0 is the free running laser emission frequency, ν is the laser emission frequency after feedback, τ_e is the laser time of flight of the external cavity, and α is the linewidth enhancement factor. As it can be proven, the behaviour of the SMI signal becomes saw-tooth like when C approaches to a value of 1, therefore allowing the use of a simple fringe counting technique to measure the displacement of a target, by accounting each transition on the signal (fringe) as a half wavelength ($\lambda/2$) displacement.

The DSMI takes advantage of the SMI fringe intervals to measure sub $\lambda/2$ displacements. This is achieved by a relatively simple comparison of two SMI signals subjected to the same reference motion/modulation. The comparison can be performed either using a double laser setup or a single laser setup. The main difference, in terms of processing, between both setups is the lack of a continuous reference in the second case, therefore, relying only in a reference measurement taken with a static target. While this might seem as a major complication, in practice both schemes have shown a similar resolution.

While the model presented in Eq.1 and Eq.2 asserts to depict the motion of a target surrounded by air, it fails to model the effects of the presence of media with different refraction indices on the laser path. Therefore a new model, which accounts for the effects of the 5 external cavities present in the experiment, was proposed (Fig. 2). By using the lasing equations it is possible to derive:

$$0 = \Delta\varphi = 2\pi(\nu - \nu_0)\tau_l + \sqrt{1+\alpha^2}\sum_{i=1}^{5}\kappa_i \sin[2\pi\nu\tau_i + \mathrm{atan}(\alpha)], \quad (3)$$

Where τ_l is the time of flight of the internal cavity, κ_i the coupling coefficients and τ_i the time of flight on the respective external cavity. Doing a similar analysis for the output power, it is possible to arrive to:

$$\Delta P \sim \Delta g = \sum_{i=1}^{5}\frac{\kappa_i}{l_c}\cos(2\pi\nu\tau_i)\,, \quad (4)$$

where l_c is the length of the internal laser cavity.

From Eq. 4 it can be easily foreseen that, for a stable setup, only the changes on the last cavity will produce the modulation on the SMI signal. This result applies as long as no change on the refractive index is produced within the third cavity since the time of flight on this cavity can vary as a function of the refractive index which can be expressed as:

$$\tau_3 = 2L_3\frac{n_3 + \Delta n_3}{c} + \tau_2 \quad (5)$$

where n_3 is the initial refractive index in the third cavity, L_3 the length of the third cavity, τ_2 the time of flight on the second cavity and c the speed of light in vacuum.

From Eq.5, two conclusions can be extracted: 1) it is possible to relate the increment of the fluid refractive index in a static fashion by measuring the difference of the initial phase shift between two SMI signals crossing the measurement media and a reference media, and 2) it is also possible to measure the dynamic variation of the refractive index as long as the dynamic is within the DSMI sampling frequency. Therefore, it is possible to equate:

$$\Delta n_3 \cong \frac{\lambda}{2}\frac{\Delta t}{TL_3}, \quad (6)$$

where Δt represents the difference found between the intervals of the measurement and reference SMI signals and T is the period between two fringes in the reference SMI signal.

Because of the limitations of the method, it is only possible to measure refractive index changes that create an apparent relative displacement lower than $\lambda/2$. Therefore, a relation between the interior diameter of the tube used to conduct the fluid, and the laser wavelength is necessary ($\Delta n_3 L_3 < \lambda/2$). On tab.1, an example of theoretical radius against the maximum refractive index change for a laser wavelength of 785 nm. It should also be considered that the full range is only applicable in the cases where we know that the measurement is steadily changing in a positive or negative change. If a signed measurement is required, the maximum measurement value is decrease by half therefore measuring in the range ($-\lambda/4 < \Delta n_3 L_3 < \lambda/4$).

Internal Diameter (mm)	***Δn_3***
20	$1{,}9625\text{x}10^{-5}$
10	$3{,}925\text{x}10^{-5}$
5	$7{,}85\text{x}10^{-5}$
2,5	$1{,}57\text{x}10^{-4}$
1	$3{,}925\text{x}10^{-4}$
0,5	$7{,}85\text{x}10^{-4}$
0,25	$1{,}57\text{x}10^{-4}$
0,1	$3{,}925\text{x}10^{-3}$

Tab. 1 Example of the internal diameter of the tube related to the maximum full range refractive index difference measurable.

In order to test the use of the DSMI in a static way, as intended for the experiments, a code simulation was created on MATLAB. The values used for the simulation are presented on Tab. 2. The reflectance values used for the simulation are estimations based on the behaviour of similar materials irradiated with a similar wavelength. As seen on the table, the refractive index on the third cavity was changed in small increments relative to the maximum measurable refractive index difference. The increment for the simulation was selected as 1/10th of the maximum measurable refractive index. Each of the signals was simulated with 125 kS.

	Value	*Units*
Wavelength	785	nm
Triangular Reference Displacement (peak to peak)	25	μm
α	5	
n_ref	1	
n1,n5	1	
n2,n4	1,55	
n3	1,33+/-9,8125e-5/2	
l_c	0,5	Mm
L1	70	mm
L2	1	mm
L3	4	mm
L4	1	mm
L5	25	mm
r_ref	0.9	
r3	0.4	
r4	0.73	
r5	0.73	
r6	0.4	
r7	0,45	

Tab. 2 List of values used for DSMI refractive index simulation.

The first thing to be noticed in the obtained waveforms (Fig. 2) is that they tend to keep a sinusoidal shape. This might be explained by the small amount of power being back-reflected from the target. This type of behaviour can also be expected in the experimental phase because of the laser scattering produced by the laser when it crosses each of the surfaces. It is also noticeable that in the case of a positive reference displacement (increase of cavity length with respect to the laser position), the detected phase change is positive for a increase of refractive index, while in the case of a negative displacement the phase change is positive for a decrease of refractive index.

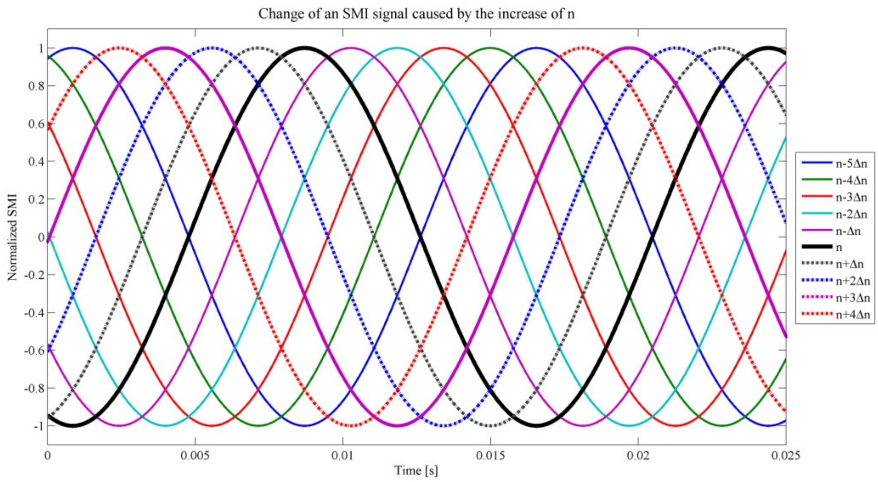

Fig. 2 Effect of refractive index change on a SMI signal.

The results obtained for this simulation are presented on Tab.3. In the first eight cases, the error can be explained by numerical effects since the minimum time differential on the simulation is in the order of $t=8x10^{-6}s$ while the period used for the SMI signal is $T=15.7x10^{-3}$, therefore limiting the resolution to $\Delta n=5x10^{-8}$. Numerical errors can also arise due to division procedures during the signal processing.

Measured Δn	***Absolute Error***
-49,1125127421999 $x10^{-6}$	50,0127421699 $x10^{-9}$
-39,2600025485024 $x10^{-6}$	10,0025485672 $x10^{-9}$
-29,4575050967527 $x10^{-6}$	20,0050966903 $x10^{-9}$
-19,6550076452251 $x10^{-6}$	30,0076452575 $x10^{-9}$
-9,8525101936975 $x10^{-6}$	40,0101936027 $x10^{-9}$
9,8024974515276 $x10^{-6}$	10,0025483452 $x10^{-9}$
19,6049949030552 $x10^{-6}$	20,0050969124 $x10^{-9}$
29,4074923548049 $x10^{-6}$	30,0076450355 $x10^{-9}$
-58,9150101937275 $x10^{-6}$	**98,1650101936626$x10^{-6}$**

Tab. 3 Simulation refractive index change measurements and Absolute Error.

In the case of the last measurement, the error can be explained by limitations of the proposed algorithm since it only compares the first period of the signal. A better way to solve the problem might be the use of a cross-correlation function instead of the proposed zero crossing algorithm. However, in both cases, the results tend to become ambiguous when the increment is close to a full period of the SMI signal. Therefore, the measurement interval should be closely taken into account to avoid possible misleading measurements.

- *Experimental work:*

The first experiment was conducted using a Hitachi HL7851G FP laser with λ=785nm output power around 30mW and injection current I_F=62.1mA. The laser was focused with a Thorlabs LA 1951-B lens and the target was placed at approximately 25cm from the lens. The mechanical reference vibration was produced by a mechanical vibrator Pasco Scientific SF-9324 which was fed by a Tektronix waveform generator with 2V and a 200Hz signal. A tube connected to a pump was used to introduce the media to be measured into position. The tube was placed between the laser and the mechanical vibrator keeping a perpendicular angle between the liquid flow and the laser beam axis, as shown in Fig.3, to reduce possible effects of the liquid velocity over the SMI signal. The SMI signal was captured using a Tektronix4041 oscilloscope. Further processing was done using custom software made on Matlab.

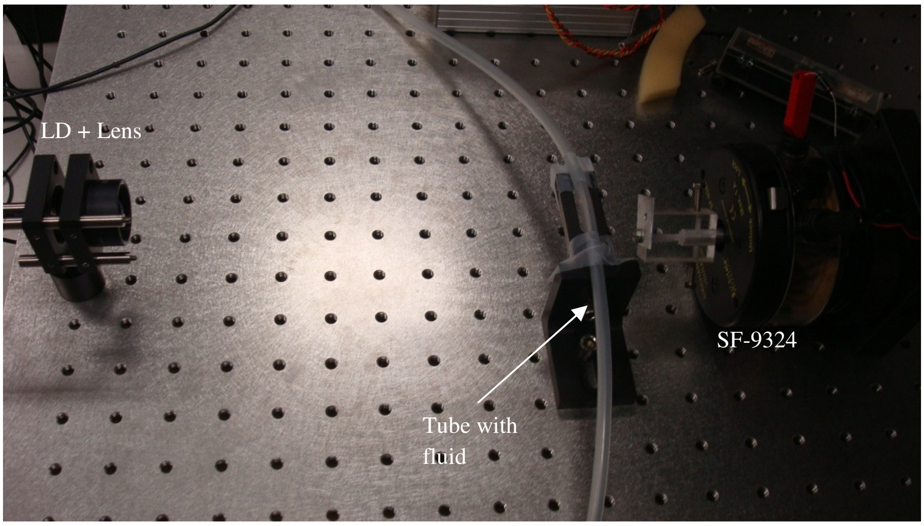

Fig. 3 Setup 1 for the measurement of refractive index change

A set of 73 measurements was performed using different media inside the tube. The first measurements recordings corresponded to air (5 samples), followed by deionized water (6 samples), a 0,1ppt (10mg/100ml) water based saline solution (7 samples), a 0,2ppt water based saline solution (11 samples) with 1MS each. The following measurements were performed using 100kS recordings to test possible differences due to sampling. The measurements correspond to: a 5ppt saline solution (8 samples), a 10ppt saline solution (9 samples), a 0,2 ppt saline solution (7 samples), a 0,4ppt saline solution (7 samples) and a 0,2ppt saline solution (5 samples).

In order to process the information, a stability analysis of the initial phase in the obtained signals was performed. The test compared the phase shifts between the different samples evaluated with the same laser at different time instants. In all of the studied cases it was found that there existed an initial phase shift which varied in magnitude and covered the full 2π range. An example of this is shown in Fig.4, which corresponds to the 10ppt solution captured with 1MS.

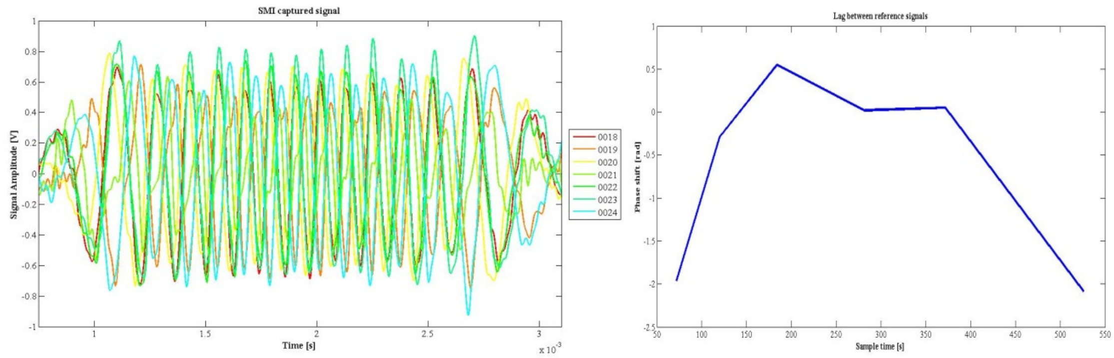

Fig. 4 Initial phase shift on 20 ppt solution SMI signals. The points in the phase shift reconstruction correspond chronologically to the increase of the number of the sample.

To try to solve this issue, in a first instance, we considered modelling the phase shift as a function of time. A new data set was collected with more samples for each different fluid condition. The results, as shown on Fig.5, did not show any time dependant behaviour. Therefore other variables needed to be considered. The first apparent conclusion was that temperature effects were acting over the laser diode. This was easily tested and observed on the oscilloscope by simply approaching the measurement setup. As soon as we were as close as 30 cm from the setup, a large variation over the SMI initial phase was observed. To reduce this issue, it was decided to introduce a laser temperature controller to the setup.

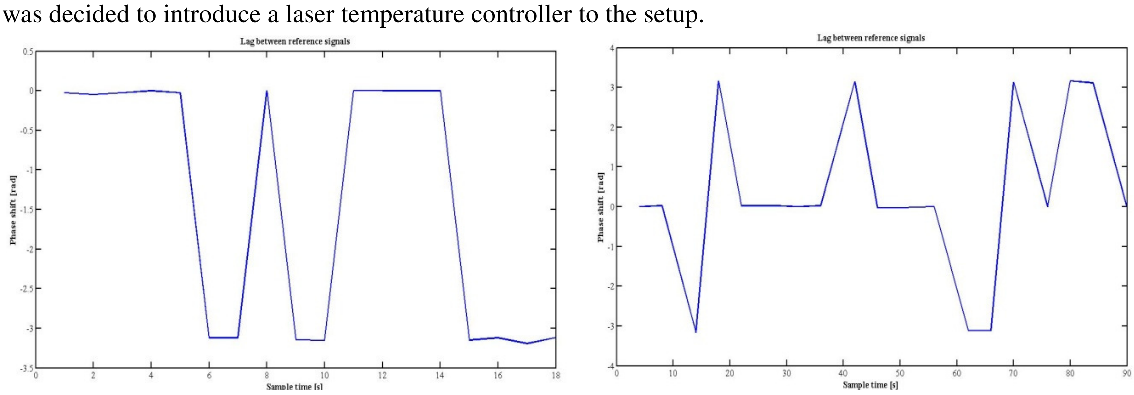

Fig. 5 Phase shift recorded for measurements on air and water media. No direct time dependency is observed.

A second setup was assembled using the same elements described on the first setup and including a Thorlabs laser temperature controller TED200c. The placement of some of the oscilloscope was changed to increase the distance to the setup and I_F was increased to 83.4mA to reduce possible instabilities due to the proximity to the threshold current. All further measurements were obtained with 1MS per signal. On a second set of measurements, the vibrator was exchanged with a PI LISA with 25µm travel length and 2nm resolution to reduce possible mechanical errors as shown in Fig.6.

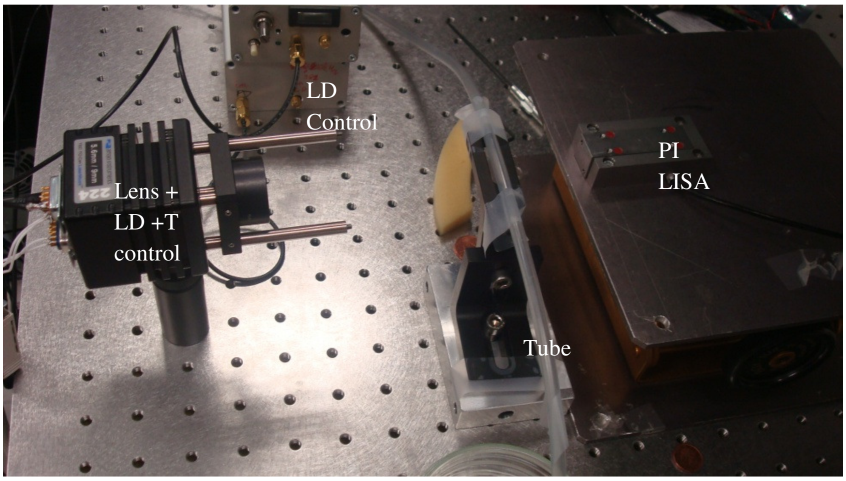

Fig. 6 Second setup.

While the behaviour of the second setup already showed a higher stability when compared to the previous setup as shown in Fig.7, some degree of initial phase shift was still observed while doing measurements in the same media. It was concluded that two possible causes might be affecting the measurements: the first was the change of temperature in the fluid due to environmental conditions and the second, the existence of sub wavelength vibrations in the tube. Because of time and material limitations, the measurement of displacement was considered as a more suitable test.

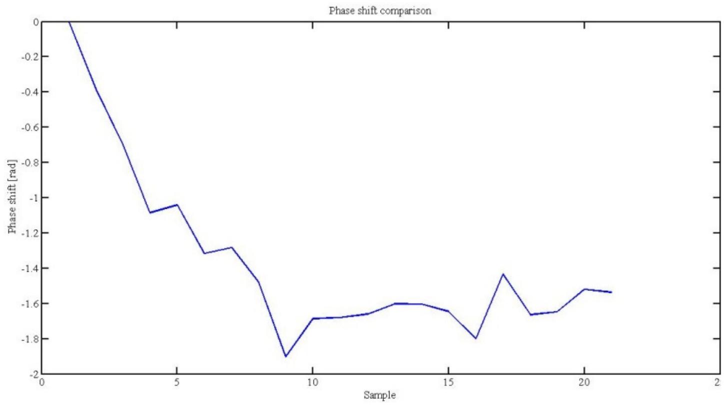

Fig. 7 Phase shift of laser measurement on the air media

All the comparative measurements were subjected to a DSMI analysis to find any possible vibration in the sub λ/2 scale. As it is shown on Fig.8, different displacements were found. Commonly the displacements were in a scale of 20nm peak to peak, however in some occasions the range drastically increased reaching values even of 100nm peak to peak. While some of this displacement can be introduced by the hydraulic pump mechanics, it rested unclear if any other mechanism was also imposing such kind of dynamics over the measuring tube. In the last day of the STSM, one of the engineers of ENSEEIHT proposed that the cause of this movement might be the piezoelectric reference displacement, which will cause a microphone-like interaction with the tube and the liquid media. This proposition seems plausible since for most of the cases, the vibration found is in the same order of frequency as the displacement frequency of the piezoelectric used as reference. A measurement trying to take into account this effect was performed and it is presented on the results section.

It is important to remark that the measurements, in my opinion, did not have enough stability to probe the technique in the tested conditions. Therefore, it will be necessary to produce new measurements in a better controlled environment; for starters, a temperature controlled environment with a control of at least 0.05C would be necessary to improve the measurements. Another possibility would be to reduce the resolution of the method by decreasing the length of the channel, thus, reducing possible temperature effects, which, in the case of water based solutions are in the range of $\Delta n/\Delta T \approx -91.52\text{x}10^{-6}$C [1].

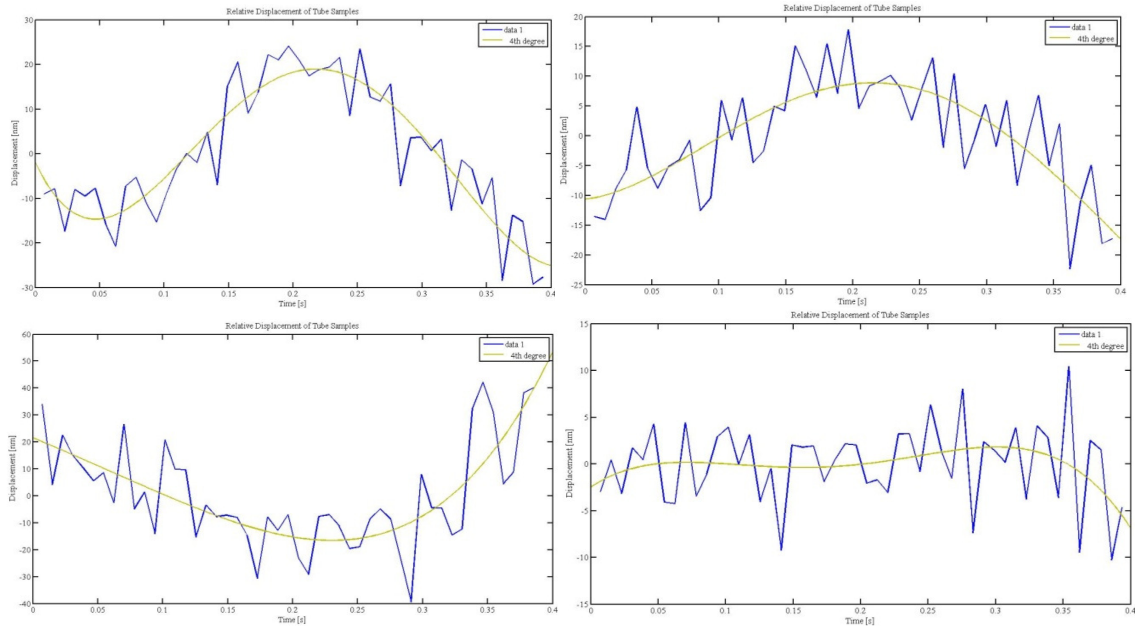

Fig. 8 Different tube displacements found during the measurement.

Description of the main results obtained:

Because of the maximum measurable refractive index and taking into account the precision of the balance used to prepare the saline solutions, it was only possible to check refractive index changes over the sampling interval. To solve this issue, we take into consideration the complete periods caused by the amount of salinity added as well as the sign of the increment. As a reference we took the values presented in [2] and take as reference the refractive index for water at approximately 20ºC for a λ=785nm as n= 1.327652281 and $\Delta n=1.333333\text{x}10^{-3}$ 100ml/gr. To ease the comparison only the expected ratios between the reference phase and the measurement phase are presented.

Comparison	***Measured Ratio***	***Expected ratio***	***Possible errors due to displacement***
Air - Water	0,0953	0,1315	+/- 0,0496
Water – 0,2 ppt Water	0,2630	0,2823	+/-0,1242
Water – 0,2 ppt Water	0,2872	0,2823	+/-0,07432
Water – 0,2 ppt Water	0,2669	0,2823	+/-0,0496
Water – 0,2 ppt Water	0,2522	0,2823	+/-0,1490
Water – 0,1 ppt Water	0,3120	0,3588	+/-0,2480
Water – 0,1ppt Water	0,3924	0,3588	+/-0,0496
Water – 0,1ppt Water	0,4665	0,3588	+/-0,1490
Water – 0,1ppt Water	0,2256	0,3588	+/-0,1242
Air – 0,1ppt Water	0,4695	0,4903	+/- 0,0496
Air – 0,1 ppt Water	0,3748	0,4903	+/-0,07432
Air - 0,1 ppt Water	0,3760	0,4903	+/-0,07432

Tab. 4 Example of measurement results

As seen on the table, the found values are not equal to the expected ratio. However, once the displacement of the tube is taken into account, most of the measurements reach the expected ratio. While the nanometric displacement can explain, to some extent, the variation with respect to the expected ratio, it is not possible to assert that it is the only cause. Also, it is important to look at the last three measurements, where the results do not reach the expected value even taking into account the displacement.

Other factors that may be affecting the measurements are the local temperature and pressure. One possible solution to this issue might be to use a temperature control box where the experiment is carried out. Also a better fixation and a more robust tube would be required to reduce the differences induced because of nano displacement. It might also be important to consider the suggestions made in ENSEEIHT to find a solution concerning the possible microphone interaction caused by the piezoelectric displacement.

In conclusion, further experiments are required to validate the proposed measured method.

Mutual benefits for the home and the host institutions:

The experience has allowed having a better understanding of the measurement of small refractive index variations with DSMI. The experience has also served as a way of sharing information related to similar research subjects and collaborations that may be performed between both institutions. In a personal opinion, the opportunity to share ideas with people that is working close to your domain is always of great benefit since it allows you to consider things that otherwise you would have neglected.

Future collaboration with the host institution:

Close collaboration between both institutions is expected to continue. From a practical perspective, the home institution can largely benefit from the expertise on SMI electronics, and SMI measurements over mili and micro fluidic circuits, while offering it expertise on a wide variety of optical systems, therefore allowing the development of more accurate and robust measurement tools.

Collaboration will also be important from the point of view of the equipment present in both laboratories. In this case, the home institution would be especially interested in the use of the micro fluidic equipment located in the ENSEEIHT laboratory.

Foreseen Journal publications or conference presentations expected to result from the STSM

The obtained results are not sufficiently conclusive to present them for publication. While the method shows some results when the samples are evaluated in short periods of time, it is not possible to affirm that no other environmental variable is affecting the measurement. In my opinion, a more controlled experiment (temperature control, change of the hydraulic pump) would be required in order to validate some of the obtained results.

Other comments

I would like to thank the COST action BM1205 for the opportunity of visiting ENSEEIHT. Also I would like to thank ENSEEIHT for their collaboration and help during the mission.

Bibliography

[1] Masahiko Daimon and Akira Masumura, “Measurement of refractive index of distilled water from the near-infrared region to the ultraviolet region,” *App.Opt* **46**(18), pp. 3811-3820 (2007).
[2] W. Mahmood bin Mat Yunus and Azizan bin Abdul Rahman, “Refractive index of solutions at high concentrations,” *App. Opt*, **27**(16), pp. 3341-3343 (1988).

STSM outcome form

STSM application number	Home institution & country	Host institution & country	BM1205 WG	Objective of the collaboration	Results of the collaboration
COST-STSM-BM1205-16456	CD6 ï UPC Barcelona Tech, Spain	LAAS France	WG4	To test differential self-mixing interferometry as a method to measure small differences in refractive index and characterize liquid media.	The DSMI method showed some potential for the measurement of refractive index changes. Further measurements are required to prove the reliability of the method. Theoretical and practical progresses on the measurement method were achieved.

I acknowledge that the described short term scientific mission was successfully carried out in the conditions here specified. Collaboration between partners was further developed and it will lead to joint work along the next months.

Toulouse, France, April 22nd 2014

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