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## DISCOVER how sustainability intersects with electrochemistry & solid state science research



### Advancements in laser-based spatiotemporal measurements of flow boiling

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Abstract. Recent advancements in laser and imaging systems, as well as in computational processing capabilities, have made quantitative optical imaging, which is often combined with laser illumination, highly adaptable, robust and reliable. Laser-based diagnostic techniques, such as planar laser-induced fluorescence (PLIF), offer the possibility of simultaneous spatiotemporally resolved measurements of temperature fields in the liquid phase at boiling conditions. In this paper, we examine the applicability of two-colour PLIF (2cPLIF), where the ratio between individual fluorescent emissions from uniformly dispersed dyes is used to take temperature measurements in the liquid phase in the presence of moving vapour-liquid interfaces typical of boiling flow. The implementation of 2cPLIF necessitates uniformity in the concentration of different dyes across the flow field. However, in the case of a multiphase flow such as boiling, thermophoresis can lead to inhomogeneous dye distributions. To overcome this challenge, a single-dye multispectral planar laser-induced fluorescence (SDMS-PLIF) method has been developed, which employs fluorescent emissions in different spectral bands of the same dye (Nile Red). The spectral characteristics of Nile Red were measured using a spectrometer to identify its temperature-sensitive bands over a wide range of dye concentrations, from 0.3to 30 mg/L. Following this, we demonstrate the measurement capabilities of SDMS-PLIF thermography as applied to a boiling flow in a miniaturised vertical square channel, gaining insight into the thermohydrodynamic interactions between vapour bubbles and a heated wall.

#### 1. Introduction

Laser-induced fluorescence (LIF) has become a valuable diagnostic tool for researchers across the globe for direct measurement of various quantities of a scalar flow field like concentration [1], temperature [2] and pH [3]. LIF is an imaging technique that is capable of extracting quantitative information by relating the intensity of fluorescence emission from dispersed dye molecules illuminated by a laser at a specific wavelength. Upon excitation, the dye molecules absorb a portion of the laser light energy and then, due to their relaxation, spontaneously radiate at higher wavelengths (lower photon energies) within a short time interval. This re-emitted

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fluorescent (incoherent and non-monochromatic) light is then recorded by an imaging system. Spectrally isolated signals of the fluorescence emission dependent only on the parameter of interest (concentration, temperature or pH) constitute the principle of measurement in LIF.

In general, for a given excitation wavelength, the choice of a dye and a corresponding band of the fluorescent light wavelengths for LIF measurements is based on the prior knowledge about the dependence of its absorption and emission spectra on variations in the magnitude of the scalar variables (temperature, concentration or pH). In the case of multiphase flow, LIF or its varieties have been conventionally employed to identify phase boundaries and liquid film thickness (e.g., Refs. [4, 5]). Meanwhile, the recent advancements by Voulgaroupolous *et al.* [6] expanded the envelope of the capabilities of LIF and PIV for the simultaneous measurement of temperature and velocity fields in pool boiling.

Specifically in the context of temperature measurement, barring few select articles, the lack of information on thermal change of the absorption and emission spectra of liquids other than water can be clearly established. As a result, most experimental studies involving LIF measurements deal with water as the most proven (but not perfect for many industrial applications) working fluid. However, the increasing number of papers on heat transfer in boiling flows of non-polar dielectric cooling liquids like hydrofluoroethers (HFEs) [7] and other refrigerants [8] dictates the need of finding suitable dyes soluble in them. This would open new avenues in engineering research by extending the applicability of these advanced laser-based measurement techniques to gain new insights into underlying thermofluidic phenomena and mechanisms of their occurrence.

Recent advancements in LIF measurements have included:

- incorporation of two (or even more) different fluorophores excited by a single laser (multiple light sources are not so common); or,
- synchronous excitation of a single dye by one or two (rarely more) lasers across two separated bands of the absorption spectrum.

Either of these options is commonly referred to as two-colour LIF (2cLIF) (e.g., see Refs. [9, 10, 11, 12]). However, these studies ignore the effect of thermophoresis that, in the case when two fluorescent dyes are used together, can be the cause of their non-uniform distribution, thus leading to violation of the intensity ratio of two signals and, therefore, erroneous measurement.

To this end, in view of the pioneering experiment by Koegl *et al.* [13], here we aim at conducting a focused study to explore the possibility of using Nile Red as a temperature-sensitive fluorescent dye for single-dye multi-spectral PLIF (SDMS-PLIF) measurements as applied to nucleate boiling in HFE-7100 fluid flowing through a vertical miniature channel. The article is split into two main parts: spectroscopic characterisation of the fluorescence emission to identify temperature-sensitive bands and discussion of the results of the nucleate boiling experiment.

#### 2. Experimental methods

#### 2.1. Formulation of dye solution

In order to compose a dye solution for the SDMS-PLIF method, the fluorophore (Nile Red) was first dissolved in ethanol to obtain its liquid concentrate (0.3 g/L). This mixture was then diluted with a predetermined volume of the working liquid (HFE-7100) to prepare final dye solutions for the flow boiling experiments. The dye concentration in the working flui for the spectrometric measurements was chosen to cover a broad range from 0.3 to 30 mg/L. Meanwhile, the experiment was performed at a concentration of C = 10 mg/L and 3.3%-vol. ethanol content.

#### 2.2. Apparatus and measurement system

The test facility employed in the present study is specifically designed to reproduce various flow boiling regimes in a miniaturised channel (Fig. 1a). The apparatus is configured as a closed liquid circulation loop. It consists of a 5 L storage tank, peristaltic pump equipped with

a coarse inline particle filter, high-accuracy mass-flow controller (maximum mass flow rate = 30 kg/h), preheaters to alter the inlet condition, optically-transparent test section, condenser, and bypass circuit where a pressure controller is installed. The test section consists of two bars of vapour-polished polycarbonate tightened together with bolts.

A square minichannel with a hydraulic diameter,  $D_{\rm h} = 5$  mm, and an aspect ratio of 1.0 is embedded in one of these bars. A heating plate made of indium tin oxide (ITO)-coated sapphire glass with dimensions of  $80 \times 10 \times 0.5$  mm<sup>3</sup> is flush-mounted at a distance of 500 mm above the channel inlet in one of the test section sidewalls (Fig. 1b). Two electrodes are attached to the electrically conductive ITO-coating using silver epoxy. At the other end, they are connected to a programmable DC power supply unit to impose a uniform heat flux ( $0 \le q \le 120$  kW/m<sup>2</sup>).



**Figure 1.** Schematics of the: (a) flow loop, and (b) configuration of the optical system used for the SDMS-PLIF thermographic measurements.

The experiments were performed at ambient pressure at mass fluxes (G) ranging from 33 to 100 kg/m<sup>2</sup>·s. The inlet subcooling temperature ( $\Delta T_{sub}$ ), defined as  $\Delta T_{sub} = T_{sat}(P_i) - T_i$ , where  $T_{sat}(P_i)$  is the saturation temperature at the inlet pressure  $P_i$  and  $T_i$  is the temperature of the bulk fluid at the inlet plenum, was varied from 5 to 25 K, and the heat flux, q, supplied to the heating plate was selected in the range from 1.8 to 15 kW/m<sup>2</sup> to achieve a nucleate boiling regime. As the present study is only aimed at demonstrating the applicability of Nile Red as a temperature-sensitive fluorophore in bubbly flows, q is limited to a maximum of ~ 15 kW/m<sup>2</sup>.

A high-speed Nd:YLF laser (Litron LDY300, 527 nm, 30 mJ/pulse, 20 kHz) was employed as an excitation source. Two high-speed CMOS cameras (Phantom VEO-E310L, 12 bit, 1280 × 800 pixels, 3200 fps, 1  $\mu$ s), each equipped with a Sigma 105 mm f/2.8 EX DG Macro lens, were placed at right angles to observe the same field of view through a beam splitter (transmission = 55%; reflectivity = 45%), as shown in Fig. 1b. The excitation light was attenuated using a notch filter (516 – 550 nm). Appropriate optical filters were additionally put on the camera lenses to pick out required spectral bands. One was a band pass filter (530-570 nm), while the other was a long-pass filter with the transmission edge at 650 nm. The laser and cameras were synchronised through a computer with the DaVis 10 software and operated at a sampling frequency of 1 kHz.

Before the boiling experiment, an *in situ* temperature calibration was taken in the test section for the SDMS-PLIF measurements. With a constant mass flux precisely controlled by the mass flow controller, the temperature of the working liquid in the test channel was slowly increased using the preheater (Fig. 1a). The temperature readings from the thermocouple inserted into the bulk fluid flow, which tip was in the field of view of the cameras, were continuously saved. After a thermal-steady state was reached, 1,000 individual frames were recorded by the two highspeed cameras, each registering the fluorescence emission from one of the two predefined spectral bands (Fig. 1b). The same calibration procedure was repeated at different temperatures. After the calibration, the mass flux and inlet temperature subcooling were set to a desired value, and the heat flux was imposed by the DC power supply in steps to achieve nucleate boiling.

#### 3. Results and discussion

#### 3.1. Nile Red spectra

In order to obtain the absorption and emission spectra of Nile Red, we employed an ultravioletvisible (UV-VIS) fluorescence/absorbance Horiba Duetta spectrometer equipped with a 4position Peltier cuvette sample tray (wavelength range = 250 - 1000 nm, working temperatures = -40 - +105 °C). Liquid test samples (HFE-7100/Nile Red mixtures) were put into Hellma quartz cuvettes (optical path length = 10 mm, total volume = 4 mL) with different dye concentrations, C, ranging from 0.3 to 30 mg/L. The temperature of the sample tray was controlled by circulating propylene glycol pumped through a thermostatic bath, while its monitoring was fulfilled by using resistance temperature detectors (RTDs) attached to it.

The temperature of the samples was increased in steps by 5 °C starting from 25 °C up to the nominal boiling point of HFE-7100 (61.2 °C at 101.3 kPa). The temperature setting uncertainty was  $\pm$  0.1 °C regardless of all other conditions. At each temperature level, the samples were held for two minutes to allow reaching an equilibrium state. At the end of the holding period, the absolute absorption and fluorescence emission spectra of the HFE-7100/Nile Red solutions were recorded for a total integration time of 50 ms at each temperature level (Fig. 2a) using appropriate charged coupled device (CCD) arrays installed inside the spectrometer opposite to the light source (75 W Xenon arc lamp) and perpendicular to the direction of the lamp radiation.



Figure 2. (a) Dependence of the normalised fluorescence emission (solid lines) and absorption (dashed lines) spectra of HFE-7100 seeded with Nile Red at a concentration of C = 3 mg/L with 1%-vol. ethanol on temperature from T = 298 to 333 K. (b) Spectrometric integrated intensity ratios between the two bands shown in (a) at different C.

#### 3.2. Temperature-sensitive bands for SDMS-PLIF

In the present study, two spectral bands, namely 550 nm  $\leq \lambda_1 \leq$  570 nm (Band-1) and  $\lambda_2 \geq$  650 nm (Band-2) were selected to maximise sensitivity of the SDMS-PLIF method towards temperature measurement (Fig. 2a). To obtain a prior understanding of the level of intensities of both bands and their dependence on temperature and concentration, values of the absolute fluorescence intensity, *I*, provided by the spectrometer were integrated across the width of each

band, overlaying the transmission function of the optical filter as measured by the spectrometer,  $\tau$ , along with the quantum efficiency profile of the camera matrix, P (Fig. 2a) as follows:

$$I_i(T) = \int_{\text{Band}-i} I(T,\lambda_i) \tau_i(\lambda_i) P(\lambda_i) d\lambda_i, \qquad (1)$$

where i is an index that takes on a value of unity (Band-1) or two (Band-2).

The conventional ratiometric 2cLIF approach implies computation of the ratio between intensities of two fluorescence signals to correlate it to fluid temperature. To this end, here we use the ratio between the integrated intensities of the two selected bands of the Nile Red emission spectrum which is evaluated through:

$$R_{12}(T,C) = (I_1(T,C))/(I_2(T,C))$$
(2)

As seen from Fig. 2a, a rise in temperature results in a consistent reduction of the fluorescence intensity in Band-2, while that of Band-1, on the contrary, increases with temperature. Thus, a ratiometric analysis of the fluorescence intensities across the two selected spectral bands on either side of the emission maximum at  $\lambda_{\text{max}}$  can be used for temperature measurement with the highest possible sensitivity, which is evidently visible in Fig. 2b.

#### 3.3. Temperature fields with SDMS-PLIF

Knowing the temperature response of both spectral bands, we can conduct thermographic imaging of a nucleate boiling flow taking place in the bespoke experimental facility shown in Fig. 1a using single-dye multi-spectral planar laser-induced fluorescence (SDMS-PLIF).



Figure 3. (a) Comparison between experimental calibration curves and spectrometric analysis with C = 10 mg/L, 3.3%-vol. ethanol. (b) Time-lapse temperature fields corresponding to the nucleate boiling regime of the vertical flow of HFE-7100 at the mass flux  $G = 100 \text{ kg/m}^2$ s, heat flux  $q = 12.8 \text{ kW/m}^2$  and subcooling temperature  $\Delta T_{\text{sub}} = 20 \text{ K}$  (spatial dimensions in mm).

In order to perform *in situ* temperature calibration, we used image intensity spatiotemporally averaged over the entire ensemble of the images and a rectangular region  $(1 \text{ mm} \times 1 \text{ mm})$  around the tip of the thermocouple. As a result, reasonably good agreement was achieved between the normalised ratios obtained from the *in situ* calibration and the spectral measurements, as seen in Fig. 3a. The fitting curve, which was derived by approximating the experimental data points, was then utilised to construct the temperature fields in the nucleate boiling flow (Fig. 3b).

It is seen in Fig. 3b that, as the bubble (marked by the yellow arrow) grows and slides upwards along the heated surface, moving away from the nucleation site, a portion of cold liquid is entrained from the bulk flow (i.e., from the central part of the channel) to the sidewall, where it mixes with hot liquid. This process is referred to as bubble-induced mixing and expected to lead to enhanced thermal transport, which is characteristic of the nucleate boiling flow regime.

#### 3.4. Limitations

The SDMS-PLIF technique has limitations inherent to conventional LIF approaches, including photobleaching of the dye, undesired reflections in the region of interest and overlapping of the emission and absorption spectra within Band-1 (self-absorption). These will lead to errors that need to be examined carefully in each case, and can potentially limit applicability, e.g., to channels with small optical path lengths, however, we do not expect these issues to be fundamentally different to those experienced in conventional LIF measurements.

#### 4. Conclusions

We report on the development and application of a two-colour PLIF (2cPLIF) method for the measurement of temperature fields in boiling flows in a minichannel. Two distinct temperature-sensitive spectral bands (550 - 570 nm and  $\geq 650 \text{ nm}$ ) were identified for Nile Red dissolved in HFE-7100. Spectrometric measurements of the absorbance and emission spectra were taken over a range of dye concentrations, from 0.3 to 30 mg/L, and mixture temperatures, from 298 to 333 K. Based on these findings and conducting a ratiometric analysis of the fluorescence emission intensities in these bands, we proposed a novel single-dye multispectral planar laser-induced fluorescence (SDMS-PLIF) technique capable of whole-field temperature-field measurements. The efficacy of the method was demonstrated in the problem of single vapour bubble formation on a heated wall in a subcooled liquid flow inside a vertical minichannel. The key features of the observed bubble-induced mixing in the nucleate boiling regime were identified through temperature fluctuation measurements in the thermal boundary layer.

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