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SCIENCE DIRECT
Experimental Thermal and Fluid Science 30 (2006) 463–472
Experimental Thermal and Fluid Science
www.elsevier.com/locate/etfs

Characterisation of liquid film in a microstructured falling film reactor using laser scanning confocal microscopy

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Received 15 October 2004; accepted 23 September 2005

Abstract

Reflection confocal microscopy was used to measure the thicknesses of falling thin films generated using microstructured plates. Each plate contained straight parallel channels through which liquid fell vertically under gravity. Three different channel dimensions were used: 300×100 μm, 600×200 μm and 1200×400 μm (width × depth). Measurements were performed for acetone, ethanol and isopropanol and gave reasonable agreement with theoretical predictions and correlations from the literature. Discrepancies were attributed to the fact that these equations do not consider parameters that affect three-phase contact lines such as surface tension and contact angle. No trend of film thickness on channel width was observed and was probably due to different surface heterogeneity and roughness characteristics amongst the plates. Measurement difficulties were encountered at low liquid film thicknesses and where the angle of the liquid meniscus was too steep.

Keywords: Microstructured reactor; Falling film; Liquid film thickness measurement

1. Introduction

Falling film reactors generate thin liquid films (typically in contact with either a flat wall or stack of pipes) that fall under gravitational pull. The thinness of these films results in rapid heat and mass transfer. As such, these systems find application in extraction, evaporation and highly exothermic processes [1–8]. Conventional falling film systems on tubes or flat surfaces generate films with thicknesses in the order of 0.5–3 mm [9,10]. A microstructured falling film reactor (μ-FFR) developed by the Institut für Mikrotechnik Mainz can generate stable films less than 100 μm thick. This reactor offers excellent heat removal capabilities and has been used safely in the direct fluorination of aromatics [11,12]. Our research aims to evaluate the performance of the μ-FFR in solid catalysed gas–liquid hydrogenations. The development of suitable catalyst incorporation methods (e.g. via alumina coatings) and reaction studies have been reported elsewhere [13,14]. This paper presents investigations of liquid film thickness and profile measurements.

A variety of methods have been employed to measure the thickness of liquid films. Examples include conductivity probes [15–17], ultrasound detectors [18], fibre-optic sensors [19], microwave techniques [20,21], laser interferometry [22,23], fluorescence [24], laser-induced fluorescence [25,26], optical measurement methods [27–29], rainbow refractometry [30] and infrared thermography [31]. The use of conductivity probes was not considered for this investigation because they require physical contact with the liquid film which would greatly affect flow characteristics due to the small flow channels and liquid film

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doi:10.1016/j.expthermflusci.2005.09.006

Nomenclature

g	gravitational acceleration	μ	dynamic viscosity
K_F	$\frac{\mu^4 g}{\rho\sigma^3}$	ν	kinematic viscosity
Re	Reynolds number $= \frac{\Gamma}{\mu}$	ν'	$\frac{\nu}{0.6\times10^{-6}}$
δ	liquid film thickness	ρ	liquid density
Γ	mass flowrate per wetted perimeter	ρ_c	density of adjoining phase
θ	inclination angle from the horizontal	σ	surface tension

thicknesses encountered. Some of the optical methods listed above were also unsuited for the system being investigated (e.g. required translucent substrates, circular surfaces).

To add to the arsenal of optical-based methods, reflection confocal laser scanning microscopy (CLSM) was employed to study this system. CLSM is a popular imaging and analysis technique that has been widely used in many areas, e.g. biology (mostly in fluorescent mode [32–35]), semiconductor materials science (mostly in reflection mode [33]) and polymer science [36–39]. The key feature of CLSM is that it has a very short depth-of-field. In conventional optical microscopes, the entire object within the lens' field of view is seen; areas at the focal point of the lens appear sharp while those towards or beyond the focal point are blurry. In CLSM, however, due to its limited depth-of-field, regions which are removed from the focal point are not seen at all due to the confocal pinhole which restricts light from levels other than the focal region from reaching the detector. The effect is similar to taking a slice through an object. By varying the depth at which the microscope is focused and scanning the beam across each layer, a succession of slices can be taken, which can then be summed to give a sharp, high resolution image of the object in question. Because CLSM collects data point-by-point on each slice, a very detailed map of a three-dimensional object can be obtained, meaning that depth discrimination and optical tomography can be performed to high accuracy [33,34].

2. Experimental apparatus and procedure

2.1. Microstructured falling film reactor

The main part of the reactor is the structured stainless steel plate for the generation of the falling film. Each plate contained straight parallel channels 78 mm long fabricated using isotropic wet etching. More details about the reactor can be found elsewhere [11–14]. Three different channel configurations were used, as shown in Table 1. In addition, a γ-alumina coated version of each plate was used as well (details of the coating procedure are given in [13] while pictures of cross sections of the coated channels are given in [40]). Slots were cut through the plates at both ends of the channels to form the liquid inlet and exit ports. Each plate measured 89 × 46 mm and was housed in a stainless steel enclosure. Liquid was fed to and removed from the back of the plate. A stainless steel gasket was placed on top of the plate. Its geometry was such that the whole length of the channels were exposed with the exception of the first and last 5 mm, which helped to distribute the liquid across the plate. The channels were exposed to ambient air.

Table 1
Channel dimensions of the microstructured plates

Plate	Channel depth (μm)	Channel width (μm)	Number of channels
Type I	100	300	64
Type II	200	600	32
Type III	400	1200	16

2.2. Confocal microscope

The experiments were performed using a Noran Odyssey video-rate laser point scanning confocal microscope, in reflection mode with a 633 nm laser. A Nikon Mplan extra large working distance (20/0.4 lens, i.e. magnification = 20×, numerical aperture = 0.4) was used. To allow the examination of the μ-FFR in a vertical orientation, the objective had to be attached to the microscope via a 90° bend (incorporating a 45° mirror) such that this lens projected outward horizontally. Because of this, the focus axis of the microscope was changed to the horizontal. As such, a microscope stage had to be constructed to allow focussing movement in the horizontal plane. It was especially important that the movement of the stage in the direction parallel to the projection of the lens could be measured accurately, as this now represented the depth at which the microscope was focused. Two screw micrometers were used to provide movement and measurement in the horizontal plane (see Fig. 1). Because of the manual set-up of this system, a restricted number of data points could be collected compared to an automated system. This led to the need for some amount of extrapolation in order to interpret the data collected. The manual data collection employed in this work resulted in experimental error of ±4 μm while confocal microscopy with high numerical aperture objectives may have sub-micron depth and lateral resolution [33].