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Experimental characterization of bubbles in liquid metals at high temperature

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1. Introduction

Due to the vast amount of CO_2 emitted nowadays through most processes dedicated to the production of H_2, the necessity of developing new technologies based on CO_2 free hydrogen generation has increased the interest in methane pyrolysis with liquid metals [1]. In this process, methane bubbles pass through a reactor filled with liquid tin, where they are converted into hydrogen and carbon. The main objective of this research is to characterize the interactions between gas and liquid tin through a process structured to overcome the different challenges found in this field such as bubble modeling and heat transfer. On account of this, the implemented study will be divided into, first, the characterization of fluid dynamics of bubbles formed in liquid metals at high temperature and, second, the characterization of heat transfer. Currently, the efforts are concentrated on the first phase, which is focused on creating an electro-resistivity probe [2] to identify the dimensions of these bubbles in liquid tin. To calibrate the probe, a prototype with water and air bubbles has been made, and optical measurements are being collected and post-processed via specialized software. The outcomes of this research will be applied in the future development of methane pyrolysis reactors to improve the performance of hydrogen generation.

2. Theoretical approach

The present study aims to characterize the bubbles generated by injecting gas into a molten tin column at high temperature. Bubble formation dynamics depend on multiple variables, including gas density, surface tension at the gas-liquid interface, column height and injection speed. The latter parameter is critical, as it determines the threshold between the formation of discrete bubbles and the transition to a gas jet, which would compromise the conversion process.

For a rigorous characterisation, dimensionless numbers linking the mentioned variables will be used to analyse the different flow regimes. The Weber number will evaluate the competition between inertial forces and surface tension, while the Reynolds number will allow the study of the relationship between fluid viscosity and gas inertia. These parameters will be explored experimentally under various conditions to define the limits of stability and behaviour of the bubbles in the reactor.

Specific ranges of bubble size and frequency have been identified in air-water systems as a function of operating conditions in previous studies [3]. However, in the case of molten tin, the dynamics differ substantially due to its high density and higher surface tension as it can be appreciated in Fig. 1. Therefore, we will start by characterising the bubble formation in the first well-studied system with water before analysing the metallic medium. The combination of experimentation and theoretical modelling will allow the characterization of bubble in liquid metals.

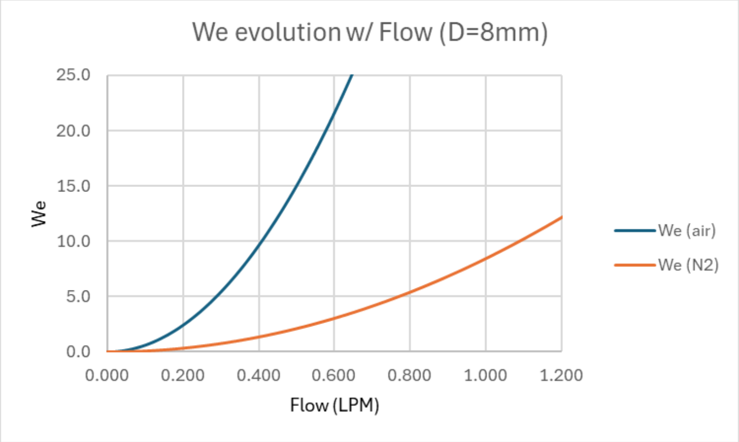

Fig. 1. Evolution of Weber number in study cases

3. Experimental setup

First, an experimental setup will be built to characterize bubbles in water. Water is chosen for initial analysis due to its relatively low complexity and the availability of relevant literature [4]. Next, the fluid dynamic behavior of inert gas bubbles in liquid metal will be examined using a high-temperature setup. Throughout the process, bubble behavior will be studied under various conditions relevant to methane pyrolysis reactors.

3.1 Bubbles in water

For the construction of the water setup (shown in Fig. 2), a transparent methacrylate tube with a diameter of 44 mm has been used. On the lower tip of the tube, a 3D-printed plate is located, whereas the other tip is opened. An O-ring is used as sealing. Thus, the nozzle can be removed, and different geometries and number of holes can be tested. The plate has a thread to join the air tube with a threaded connection.

Air blows from the bottom of the tube through the nozzle. To measure the pressure and the flow rate, a flowmeter and a manometer have been placed before the inlet. Both magnitudes are controlled by a valve. Water setup is fed with an air flow rate from a compressor able to work from a gauge pressure of 0 to 8 bar, although water columns higher than 250 mm cannot be reached in this setup due to its size and gauge pressure higher than 0,5 bar is not required.

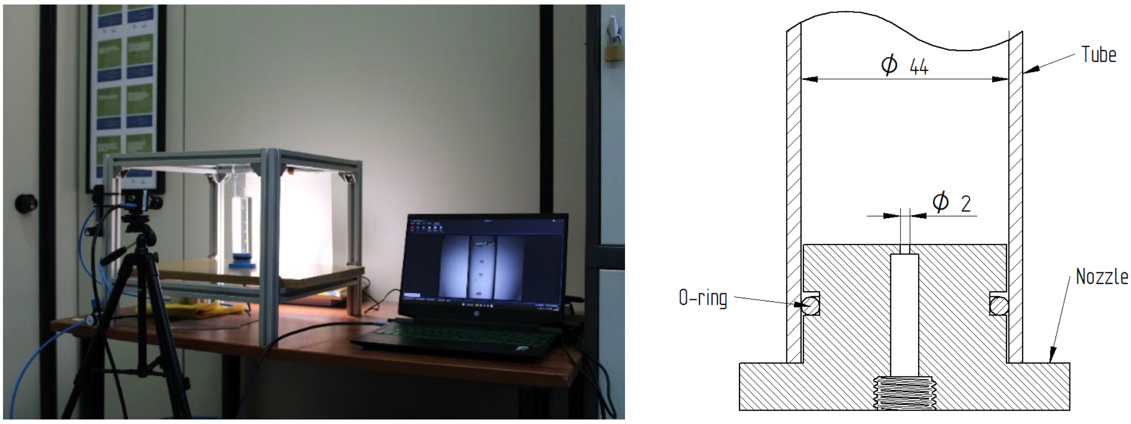

Fig. 2. Water setup

Regarding the camera setup, an industrial camera (DFK 37BUX178) with a frame rate of 60 fps and a resolution of 3072x2048 is being used to record the bubbles produced. Attached to the camera is a lens (FJN-HF16XA-5M) with a focal length of 16 mm and manual focus. The camera is positioned next to the water setup on a tripod, which allows to accurately focus the image on the bubble column. Behind the water setup, there is a LED light source with a diffuser panel in front of it, ensuring the light is smooth and does not interfere with the videos taken. The light source is necessary to produce diffraction on the bubbles surface, which will later allow the software to identify the bubbles outline.

A software written in MATLAB is used to identify the size and speed of the bubbles through image processing. The software can analyse individual frames of a video to detect the presence of bubbles and characterize them. Firstly, the frames are processed into a binary image to easily identify the bubbles shape. This involves converting the grayscale image into a binary format, where the bubbles distinguish from the background. Afterwards, the size of the bubble is measured by scaling to reality the number of pixels along the mayor axis. To calculate the speed of the bubbles, the software measures the frequency at which bubbles pass through a specific area. This is achieved by tracking the bubbles in consecutive frames and by calculating the time between the appearance of different bubbles. An screenshot of some of the results of this software is shown in Fig. 3.

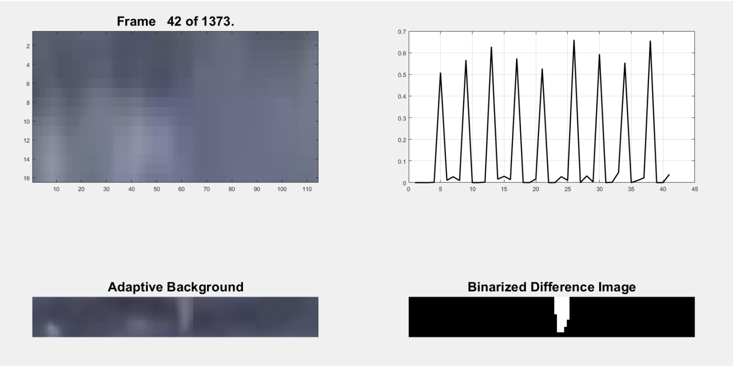

Fig. 3. MATLAB results on frame processing

3.2 *Bubbles in tin*

The high-temperature setup is shown in Fig. 4. It consists of a 50-millimetre quartz crucible covered by an high-temperature truncated-cone silicone lid, pierced to access the entrance of N2 gas flow in the crucible, through a 2-millimetre internal diameter U-shape tube, connected to flow and pressure regulator valves, all of which generate bubbles inside the tin. Additionally, a stainless-steel tube facilitates the gas exit. The crucible is introduced in a ceramic band heater and wrapped in insulating ceramic coating to reduce heat loss. To melt and maintain the liquid metal under optimal thermal conditions, the entire experiment is being surrounded by stacked insulating bricks. The temperature measurements are taken by implemented thermocouples. The power supply delivers 300 W, heating the quartz walls to nearly 500ºC to ensure the tin melts (melting point: 231.9ºC). Special attention is given to maintaining a nitrogen atmosphere throughout the experiment to prevent oxidation of the tin, which has occurred in previous tests, ad tin oxide is challenging to remove from the quartz

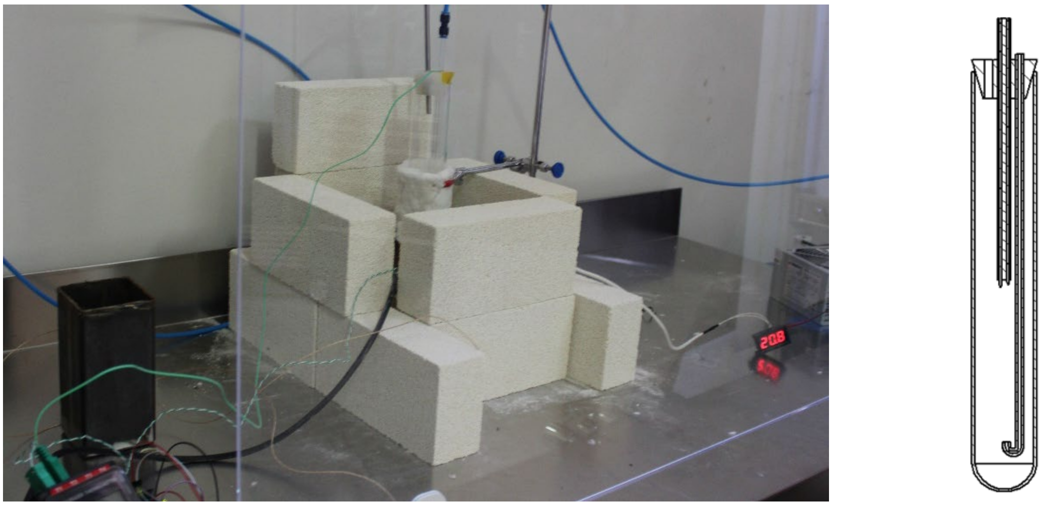

Fig. 4. High-temperature setup with tin

3.3 *Electro-resistivity probe*

Since bubbles cannot be directly observed in the high-temperature experiment due to the opacity of tin, alternative methods must be used for characterization. In this case, an electro-resistivity probe is being developed to measure bubble size and velocity in the tin reactor. The probe will first be tested in water and calibrated against optical measurements from the camera. This calibration will then inform the design of a high-temperature-resistant probe for the experimental setup. A diagram of the electro-resistivity probe's electrical circuit is shown in Fig. 5.

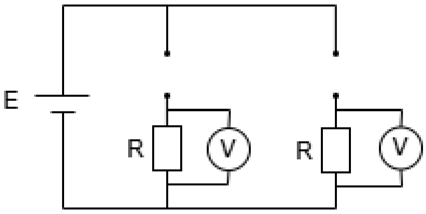

Fig. 5. Diagram of the electro-resistivity probe's electrical circuit

Three wires are connected to the power supply: two of them are going to detect the flow of bubbles by opening and closing the circuit, while the third wire is the ground. A resistor and a voltmeter are connected to the circuit and an Arduino board to record the size and speed of bubbles. This probe takes advantage of the high difference between the resistivities of the air and water: while there are not any bubbles, the circuit is closed and voltmeters measures the voltage; when a bubble passes through the uncovered tip of the wire, the circuit is opened and the voltmeter measures 0 V. Since the tips of the wires are placed at different heights, two different signals are received, and the speed and size of bubbles can be estimated.

As the tin reactor reaches high temperatures, chemical reactions between the liquid metal and the electro-resistivity probe may occur. To prevent it, noble metals electrodes such as tungsten are used to conduct electric current. Moreover, they were recovered by a heat-resistant alumina cylinder tube to maintain its distribution and reduce overheating as shown in Fig. 6.

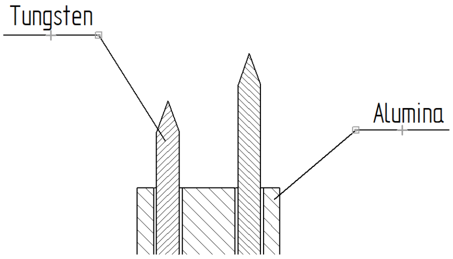

Fig. 6. Diagram of the electro-resistivity probe

4. Results

The setups for both tin and water are now operational, and tests of the electro-resistivity probes are underway. The results will be presented at the conference.

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