A FACILITY FOR THE EXPERIMENTAL ANALYSIS OF GAS BUBBLING BEHAVIOUR INTO LIQUID METAL COLUMN FOR THE DESIGN OF CHEMICAL REACTORS

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ABSTRACT

The application of liquid metal technology into the chemical industry is very limited up to now. Nevertheless, liquid metals are very interesting fluids for their high thermal conductivity and diffusivity, and stability at very high temperature what implies very high boiling temperatures and low vapor pressures. The reasons may be based on their low compatibility with steels due to high corrosion rate. Moreover, the need for the development of very high temperature processes for the clean, circular economy has attracted the attention to new innovative reactor proposals that could operate far beyond 1000 ºC. In particular, it was tested successfully a proof-of concept of a liquid metal reactor for methane consisting of a liquid tin column reactor with a porous inert bed(Geißler et al. 2015). To advance towards the industrial development of such concepts, engineering tools and design principles has to be qualified for the scaling of liquid metal reactors. We are building an experimental device for the qualification of bubbled dynamics of gas injection into a liquid metal column, including the implementation of porous media, and a scale that could serve as to evaluate the multi-phase dynamic behavior at high temperature reactors. The facility will be presented, and some first lessons learn and data analysis of its operation.

1 INTRODUCTION

Almost any added value process is based on a material transformation strongly dependent on how energy is applied and which level of temperature would be achievable. Exergy is in fact one of the most important thermodynamic property defining the quality of the energy to be used in chemical, industrial and power process and facilities. The efficiency of the direct utilization of thermal energy is increasing with its exergy content, mainly derived from the temperature that is feasible to running a process. The traditional example is the mechanical transformation of enthalpy in a thermodynamic cycle following ideally the Carnot law. The efficiency of such process in power plants is limited by the maximal temperature of the cycle fluid (Alfellag 2017) (steam, supercritical steam, or gas/air). The same may apply to chemical reactions, which capacity of heat to promote them depends on its exergy (temperature) content. Electrochemistry is in fact a mean to promote chemical transformations by the application of high energy carriers, as it is the case of electrons.

Materials is one of the most limiting boundaries to operate at very high temperature, as they must keep structural and physical integrity, as well as resistance to corrosion in the case of using a heat transfer fluid. The capacity of heat transfer fluids as air or water is limited, either by their density and thermal diffusivity, or by limited operation temperature. The need to extend beyond the current limits opens the door to consider alternatives as liquid metals (Lorenzin and Abánades 2016). They offer high density, thermal capacity and diffusivity; and many of them very high boiling temperatures and low vapor pressures. As drawback, high temperature operation amplifies their corrosion impacts on usual structural materials, mainly steels.

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As described, their good cooling capabilities are the reason for their proposal of liquid metals in nuclear reactor engineering (Subbotin et al. 2002), aerospace , solar energy and industrial waste heat utilization. As an example, sodium has been tested as coolant/moderator in fast nuclear reactors (Spencer 2000), or as coolant of solar thermal central tower receivers (Arévalo and Abánades 2023). Other additional features of liquid metals are high volumetric expansion coefficient (high Grashoff number-strong natural convection), and low Prandtl number (turbulent effects have relatively less importance that heat conduction in the flow).

In spite of their good thermal-physical properties, the utilization of liquid metals for the design of chemical reactors has been neglected by the research community (Daeneke et al. 2018). The reasons may be based on their low compatibility with steels due to high corrosion rates (Shin et al. 2012). Nevertheless, the application of liquid metals in chemical engineering as reaction media is being proposed as an alternative for high temperature processes for their potential for a stable performance in a wide range of temperatures. For instance, tin has a low vapor pressure ($5{,}78\cdot 10^{-21}$ Pa @ 505 K) and large liquid-phase temperature range (232-2602 ºC). One of the process that would play a role in the transition towards a decarbonized future is hydrocarbon pyrolysis (Clarke and Abánades 2021), that transform hydrocarbons into solid carbon and hydrogen in the absence of oxygen.

There are different techniques to produce hydrocarbon pyrolysis (Patlolla et al. 2023). In particular, liquid metal driven methane pyrolysis has been proposed as a potential technology that could lead to the practical industrial implementation of methane cracking (Abánades, Rubbia, and Salmieri 2012) to produce carbon and hydrogen in the same process. Its main characteristic is the management of the carbon particles into the reactor by differential density between carbon and liquid bath, and the scaling capabilities of high thermal diffusivity of a liquid media. Liquid tin or gallium(Leal Pérez et al. 2021) are proposed as molten media for the reaction, as well as liquid metal alloys containing nickel, with a catalytic effect (Upham et al. 2017). Additional work should be done to determine the scalability of this concept, and the compromise between complexity, performance and economics of the process from the utilization of various liquid metal, with or without embedded catalysts. Corrosion issues have to be addressed for the evaluation of the lifetime of large-scale reactors. Recent experiences with liquid tin at high temperatures and making use of ceramic materials did minimize corrosion concerns (Y. Zhang et al. 2018). Previous work (Abánades-Velasco and Martínez-Rodríguez 2023) has concluded that some technological uncertainties should be solved for the reliable up-scaling of the liquid-metal pyrolysis process as fluid-mechanical models, carbon extraction systems operating at high temperature. In this communication, we will describe the status of a facility that we are building to evaluate fluid-mechanic models for gas bubbling into liquid metals and evaluate parameters as liquid metal hold up, or bubble ascending velocity, critical for scaling liquid metal reactors.

2 GAS INJECTION INTO A LIQUID COLUMN

As described, some concepts are under development related to gas processing into a liquid media. Such processes are based on the injection of a low-density gas phase in a high-density fluid which naturally implies the motion of the gas phase by buoyancy. In a liquid column, and in the particular case of liquid metals, (Kazakis, Mouza, and Paras 2008) reported that the terminal velocity of gas bubbles may be formulated as for Equation (1).

$$v_b = \sqrt{\frac{4gD_b(\rho_{lm} - \rho_g)}{3C_D\rho_{lm}}} \tag{1}$$

Being D_b the diameter of the bubble, ρ_{lm} and ρ_g the density of tin and the gas, and a hydrodynamic coefficient (Cd) of the form described in Equation (2).

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$$C_D = \frac{21,12}{Re} + \frac{6,3}{\sqrt{Re}} + 0,25 \qquad \text{For } 0,02 < Re < 200 \tag{2}$$

There are no many confident correlation for the evaluation of the diameter if a bubble when injected through an orifice or a porous, except for previous work experimentally tested for injection trough a porous sparger in water provided by (Kazakis, Mouza, and Paras 2008).

Bubble evolution into the liquid media is reported as dependent on the Weber number, either at injection or at the column as described by (Hu et al. 2016). Some other analysis have reported bubble breakup due to the kinetic forces when ascending in a liquid flow and its dependence on a critical Weber number (Eskin, Meretskaya, and Vikhansky 2020). Definition of the Weber number may be based directly on the kinetic velocity respect to the fluid viscosity defined by the density of the liquid (ρ_l), its velocity (v), the bubble diameter (D_b) and de surface tension, or by the turbulence parameters as the kinetic energy dissipation rate (ε):

$$We = \frac{\rho_l \cdot v^2 \cdot D_b}{\sigma} \sim \frac{2\rho_l (\varepsilon D_b)^{2/3} D_b}{\sigma} \tag{3}$$

At high Weber number > 1, inertial forces are dominant and the bubbling regime flow is transformed into gas jets (T. Zhang, Wu, and Lin 2020), what drives to much faster velocities.

The evaluation of the type of flow and parameters as the terminal velocity is very relevant for the evaluation of chemical reactions with low kinetics. Understanding the flow regime is critical for the coupling between the chemical and fluid-mechanics tools.

For the case of the utilization of a liquid metal media, with thermal-physical properties leading to low Prandtl numbers, design tools are less reliable than in the case of common fluids as water or air. In addition, many chemical processes involve multi-phase multi-component flow (gas components, liquid metal and particles) that need to be coupled with chemical kinetics. Regarding thermal-hydraulics, two-phase flow CFD analysis in liquid metals has been extensively done in the past two decades for nuclear lead-cooled reactors and accelerator driven systems(Wang et al. 2021). For multi-phase flow involving bubble evolution, most of the research reported is based on Volume of Fluid (VOF) methodology and k-ε turbulence model in combination with commercial codes as ANSYS-FLUENT and OPENFOAM, even applying porous models (Abánades and Peña 2009; Chen et al. 2017). From our previous analysis (Abánades-Velasco and Martínez-Rodríguez 2023), some issues must be addressed to tune the application of low Prandtl number fluids to chemical reactors:

- Bubble and their evolution in pools or channels. Evaluation of their flow regime.
- Validation of simplification assumptions for solving the Navier-Stokes equation (RANS), to grant reduction of computational power to implement direct simulation models (DNS)
- A better qualification and validation of CFD codes with low Prandtl number (Pr) fluids such as liquid metals to estimate heat convection/conduction transfer phenomena.
- Improvements in the static porous bed treatment models.

The upscaling of liquid metal chemical reactors requires the development of suitable tools that could estimate with good accuracy the performance of gas bubbling and jet evolution in a low Prandtl liquid media with multi-phase flow. For this purpose, dedicated infrastructures and experimental devices should be build to provide data for the qualification of engineering simulation tools, adjusting models and hypothesis to simplify those tools as to grant its practical implementation and use in industry.

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3 MODEL FOR EXPERIMENTAL VERIFICATION OF GAS BUBLING CHEMICAL REATORS

In the framework of the BRAINEN project (www.brainen.es), we are developing a facility for the experimental verification of fluid-mechanic phenomena of interest for chemical reactors. In particular, our aim is to contribute to shed some light about which are the impact of design options as the case of the implementation of packed, porous bed to the dynamic lifting of multi-component gas into a liquid metal column.

3.1 Design of the facility

The facility has been designed for experimental checking of fluid-mechanic parameters regarding the injection of gas into a liquid metal column. The apparatus should be able to provide valuable and quality data for the upscaling of future liquid metal reactors proposed for chemical processing. This is the case of methane pyrolysis (Upham et al. 2017), a process that converts hydrocarbons into solid carbon and hydrogen. Such type of processes operates at high temperature (> 500 ºC) enhancing their chemical kinetics by catalysts, or by the increasing liquid metal/gas temperatures reaching up to 1200 ºC. The evaluation of the residence time of the gas phase flowing through the liquid metal media plays a fundamental role in the design of this reactors. Therefore, the experimental model should be able to generate datasets for the development of engineering tools to scale such reactors with a clear estimation of aspects as the processing gas flow rate, rising velocity, impact of packed beds in the rising column, temperature homogeneity, or set-points for an optimal flow regime. The facility scheme is shown in the Figure 1.

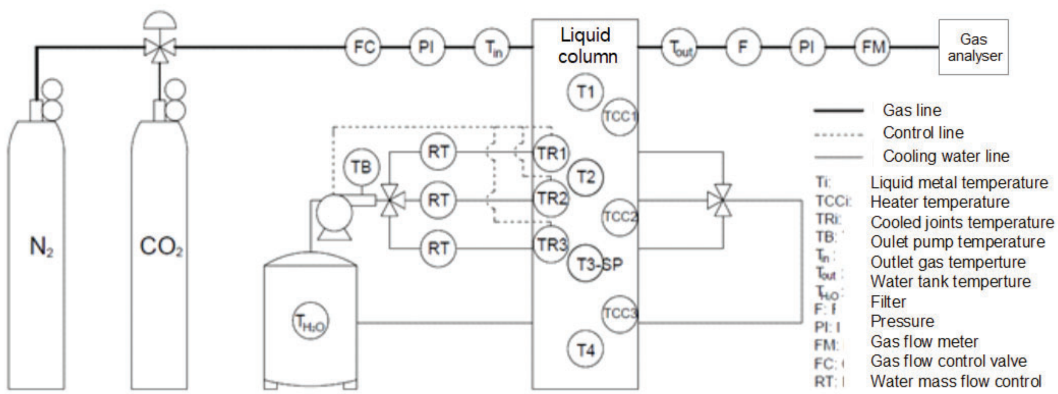

Figure 1: Sketch of the facility.

The facility is designed to operate with liquid metals at high temperature. One of the main challenges was the selection of suitable structural materials, which should have low corrosion rates. We selected Tin as main liquid metal bath to fill the column based on its wide liquid phase range with low melting point (231.93 ºC) and a very high boiling point (2602 ºC), as well as low vapor pressure ($5{,}78\cdot10^{-21}$ Pa a 505 K). The facility is instrumented as to evaluate the following data:

- Gas residence time: a gas analyzer is placed at the outlet of the gas circuit of the facility to evaluate changes in composition, that will be one input to estimate residence times for each gas by the detection of controlled changes produced at the gas inlet, combining inert gases as nitrogen and carbon dioxide.

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- Bubble formation: a set of transducer SW-PC300 are located to evaluate the pressure oscillations that will be produced by bubble detachment inside the liquid metal column, as described by (Sun et al. 2020).
- Transition from bubbling to jet regime: a combination of pressure and residence tine in the reactor will show the critical Weber number and the impact of the regime in the residence time.
- Temperature homogeneity: The set of K-type thermo-couples will show the temperature profile in the liquid metal column.
- Thermal losses will be monitored by external thermal-imaging cameras of the external insulation surface, to evaluate thermal efficiencies in combination to the regulation of the electric coils that will provide heat.
- Evaluation of the effect of packed-bed in the liquid metal column.

The facility is specially conceived for the test of gas bubbling into liquid metal baths. Material compatibility with wide liquid range of proposed metals (Lorenzin and Abánades 2016) as tin, lead, eutectic lead-bismuth, gallium or gallinstan should be granted. Such materials are generally very corrosive, especially with steels, so that they dissolve some components such as nickel or iron (Simon, Terlain, and Flament 2001), even at moderate temperatures (< 600 ºC) what bans its use for high temperature chemical reactors. An option is quartz that is very resistant to tin corrosion but the fragility, the cost and the manufacturing are the weak points. It is known that the use of high temperature resistant metallic materials in contact with liquid metals can only be approached by applying surface treatments that protect the steel, for example, with oxidation coatings, or by layers of carbides or nitrides. There are other metals such as tungsten or rhenium that can be used, but their costs make them unfeasible as a realistic alternative. Corrosion rates have been reported to be greatly reduced in composites with ceramic materials such as alumina, silicon carbide, mullite or graphite(Y. Zhang et al. 2018). For our device we have selected silicon carbide for design basis.

The experimental infrastructure will provide very valuable data for the qualification of design codes and guidelines for liquid metal chemical reactors, as well as a testbench for material performance in long-term operation under realistic conditions.

3.2 Evaluation of experimental parameters

An EES 1-D model (Klein 2021) has been developed for the sizing of the facility, evaluating mass and energy flows. The tool for the design of the liquid metal column has to show the effect of the main uncertain parameters and its influence on the operation of the experimental apparatus. One of the most important is the gas hold-up, that should be kept under control to liquid metal level into the column, avoiding unexpected operational events as liquid metal leakage of intrusion onto the gas circuit. The model allows to re-evaluate the design to adapt to the requirements of the manufacturer in order to keep cost under the budget constraints.

The device is composed by a cylinder defined by an internal diameter and length. The selection of those design features is based on technical criteria within the availability of a manufacturer to supply a tube at reasonable costs. Our aim is to provide an infrastructure to shed some light about the fluid-mechanic phenomena at scale. For such reason, the diameter of the column has been intended to minimize wall effects on the general movement of gas bubbles or jets.

There is scarce information reported about the size of bubble diameter into liquid metals or opaque fluids. (Sun et al. 2020) reported comparison of correlations and experimental data. A correlation for the estimation of the formation of bubble diameter has been developed by (Jamialahmadi et al. 2001) and described in

$$\frac{D_b}{D_o} = \left[\frac{5{,}0}{Bd_o^{1{,}08}} + \frac{9{,}261\ Fr^{0{,}36}}{Ga^{0{,}39}} + 2{,}147\ Fr^{0{,}51}\right]^{\frac{1}{3}} \tag{4}$$

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Equation (4) provides the ration between the bubble diameter (D_b) and the injection orifice diameter (D_o) depending on the Bond (Bd), Freud (Fr) and Galileo numbers (Ga) defined in equation (5) as:

$$Fr = \frac{v}{\sqrt{gD_o}} \qquad Ga = \frac{gD_o^3}{\nu^2} \qquad Bd_0 = \frac{\rho_L g D_0^2}{4\sigma} \tag{5}$$

From such work, bubble diameter is estimated to be on the order of a few mm. To allow the analysis of various orifices with a potential individual bubble detachment and further bubble coalescence, a device diameter of more that 10 cm has been considered.

A sketch of the facility is depicted in Figure 2, with the gas injection at the bottom trough a set of orifices, a central cylinder and some functional sections at the top to avoid liquid metal losses by a cooled condenser. Some liquid metal may be toxic by inhalation, and operating at high temperature may cause the formation of liquid metal vapors. The condenser, located in a cooled section far from the heating section of the column, avoid the gas stream to sweep along liquid metal vapors.

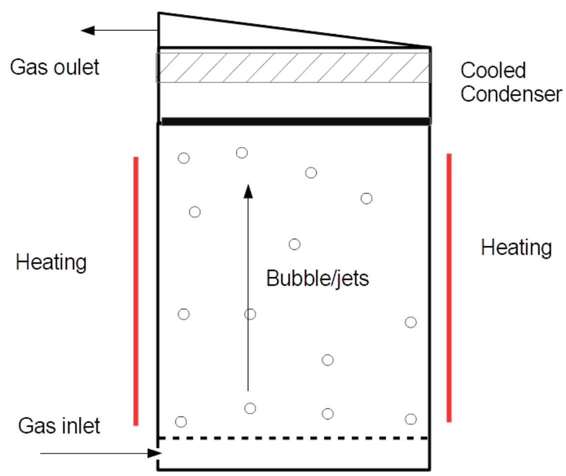

Figure 2: Basic model of the liquid metal column.

The main design parameters of the facility are listed in Table 1. The central cylinder length is fixed to 1.5 m, to provide enough height as to test different gas flow rates, even with high hold-up, to evaluate the impact of high gas-to-reactor volume processing. As an example, Figure 3 depicted an analysis of the liquid metal level increase versus the gas flow rate that is processed in the facility. The analysis allows to modify the gas and liquid metal species, in this case it is shown the case of tin and methane.

Additionally, either packed-bed, different types of filling structures, or just liquid metal may be tested. Depending on the type of column, the tin (as reference metal for this facility) mass could be loaded in a range between 50 and 200 kg. The design maximum heating power based on electric heaters is 15 kW, to allow fusion of the liquid metal in a reasonable time, and reach temperatures up to 1200 ºC.

The numerical model 1-D model has been used as well for the evaluation of the insulation layers to keep the external wall temperature within safety standards (60 ºC). The insulation is based in layers of high temperature silica bricks and fibres and microporous plates, with a total thickness of 33 cm. That model has allowed to evaluate design parameters that will validate the performance of the injection. **Figure 3** shows the expected bubble diameter respect to the size of the orifice, that has been fixed to 0.6 mm.

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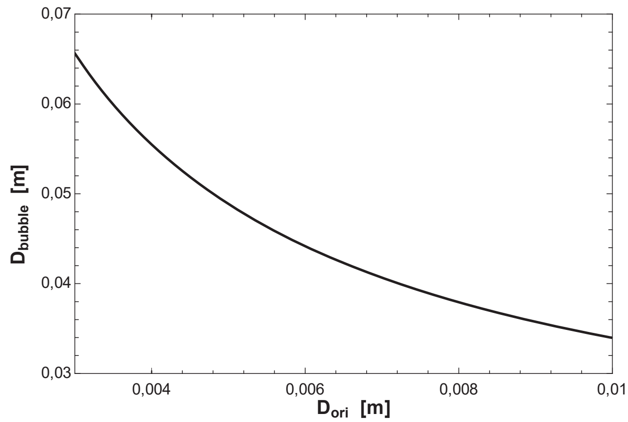

Figure 3: Bubble generated depending on the injection orifice diameter.

Table 1: Design parameters of the liquid metal column.

Parameter	**Value**	**Unit**
Height	1,5	m
Internal diameter	17	cm
Gas injection diameter	0.6	mm
Number of orifices	40	
Sn mass	50-200	kg
Heating power (max)	15	kW
Thermal losses	675	W
Gas flowrate.	0.15-1.2	Nm^3/h
Temperature (max)	1200	ºC

3.3 Instrumentation

The experimental infrastructure will be instrumented for the evaluation of the most important fluid-mechanic parameters that would allow to qualify design codes for liquid metal chemical reactors. Sensors will include:

- Thermocouples inside the liquid metal column and at the surface of the main cylinder. Will provide data for the evaluation of the thermal homogeneity and control of the heating coils. Type K
- Thermocouples at the facility joints, to check thermal losses at the joints and keep the integrity of the sealing gaskets. For this lower temperatures conventional PT100 thermocouples are used.
- Pressure transducer, to help evaluation of bubble detachment and liquid metal level.
- Gas analyzer at the end of the gas circuit to detect changes in gas composition, either for future chemical evaluation or for tracing gas mixture modifications. Model GenTwo V2.4 Multigas Analyser
- Mass flow meters.
- Electric power delivered to the heating coils for efficiency and losses evaluation, up to 15 kW. 3-section electric heating.

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4 RESULTS

We have presented the basic design of a facility for the experimental. The facility is already decommissioned and the final installation is in progress. The device is located at the UPM campus in Tecnogetafe in an open space where a standard container is used as control room, as shown in Figure 4. Such sketch shows the gas tanks for the injection into the device, as well as a couple of water tanks for cooling purpose of critical parts, as it is the case of joints and instrumentation. A 3-D view of the apparatus is shown in Figure 5. The heating system and the rest of the parts has been designed to allow different configurations and maintenance.

The analysis of the raw data (temperature at several locations, pressure fluctuations, gas flow rates and composition) is expected to provide information for the validation of the fluid-mechanic phenomena and tools related to gas injection into a liquid metal column. Our commitment is to test various gases and liquid metals.

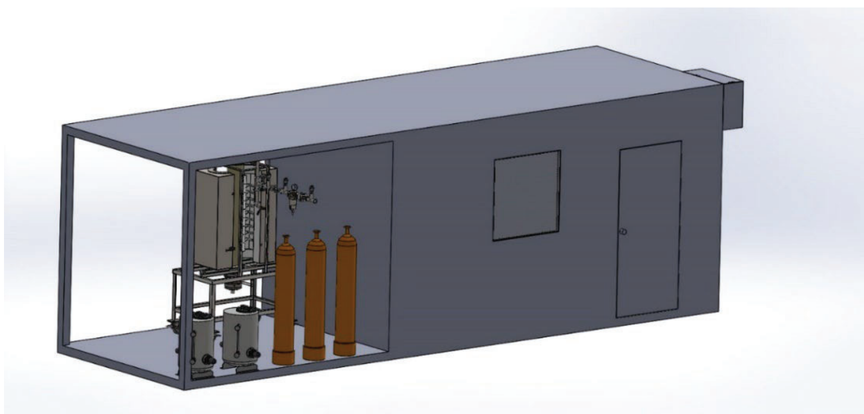

Figure 4: 3-D representation of the experimental facility.

Figure 5: 3-D representation of the experimental model.

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We have done a preliminary evaluation of some features (Figure 6), one of the liquid metal level (gas hold-up) respect to the gas injected in the device. This will be a very valuable information to analyze the residence time of the gas in the column.

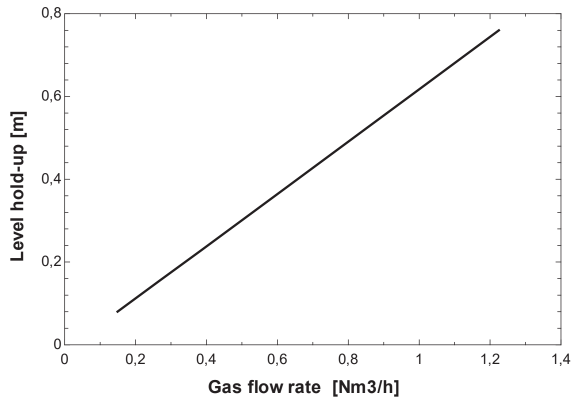

Figure 6: Gas hold-up versus gas flow rate in the facility.

5 CONCLUSION

We have presented in this communication the design and status of the construction of an experimental facility for the evaluation of gas injection into a liquid metal column. We have identified that is a need for the development of new high temperature processes in the chemical industry. Those processes include gas processing, as hydrocarbons, for its decomposition, as well as the treatment of other compounds that would need high temperatures for its processing, as solid oxide reduction. Some metals are ideal media for high temperature processing for their broad range of liquid phase, as it is the case of tin, lead, or even mercury. Nevertheless, there are very few data available for engineering tool qualification and validation, specially for multi-phase, multi-component phenomena with a variety of chemical species.

The facility that we are building expects to operate with a variety of liquid metals gases and it will start operation in due term, providing experimental verification of phenomena as the bubble-to-jet transition on the injection of gas into low-Pr number fluids, gas flowing trough packed beds in high viscosity fluids, or the scaling laws for liquid metal reactors.

NOMENCLATURE

d	diameter	(m)
ε	dissipation rate of the turbulent kinetic energy	(m^2/s^3)
Fr	Freud number	(-)
g	gravity constant	(m/s^2)
Ga	Galileo number	(-)
ρ	density	(kg/m^3)
σ	surface tension	(N.m)
Pr	Prandtl number	(-)
We	Weber number	(-)

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Subscript

b bubble
l liquid
o orifice

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