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Research article

Mechanical stirring: Novel engineering approach for in situ spectroscopic analysis of melt at high temperature

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ABSTRACT

This paper proposes a novel engineering approach to control molten metals at high temperatures considering the industrial environment of such materials. To reduce analysis time and cost, in-line analysis techniques are more advantageous as they provide real-time information about melt composition. For this reason, recent research works focus on the development of new devices based on LIBS (Laser Induced Breakdown Spectroscopy). These devices allowed for analyzing impurities inside molten metals with great performance. However, improvements related to the immersion probe conception are still required. Indeed, the previous design used bubbling inside the melt, leading to spatial instabilities of the surface analyzed by LIBS. The solution presented here is mechanical stirring by innovative rotary blades which will be a part of an immersion LIBS probe. Their rotation will generate a representative, renewed, and stable surface that will be targeted by spectroscopic techniques in general and particularly by LIBS laser for molten metal monitoring at high temperatures. This solution was validated using experimental tests based on particle imaging velocimetry (PIV) in water at room temperature and then applied to silicon melt at high temperatures. To do so, it was necessary to design a system that allows the introduction of the blade in the melt and controls its rotation.

1. Introduction

The control of impurities concentrations in the molten metals is generally realized using ex-situ techniques of characterization on solid-state samples such as ICP-OES (Inductively Coupled Plasma Optical Emission Spectroscopy) or GDMS (Glow Discharge Mass Spectrometry) because of their high sensitivity. However, the use of these techniques is limited by the analysis delay and cost. In-situ techniques can provide real-time analysis of the melt composition. Recent research works focus on the development of new devices and tools coupled with LIBS (Laser Induced Breakdown Spectroscopy). This latter is a remote and fast-spectroscopic technique that can be applied to any material with a very low detection limit (ppm). It consists of focusing a laser on the analyzed material surface that will generate a micro-plasma [1]. The spectrum emitted by the micro-plasma analysis will allow the determination of the nature and the concentration of the elements present in the material [2–4].

Recently, the application of LIBS to metallurgical melts knows an increasing interest. Indeed, among the first users of LIBS for controlling metallurgical melts, Paksy et al. [5] focused their work on signal optimization and normalization of liquid aluminum

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samples. Later, Gruber et al. [6,7] applied LIBS to a steel bath for monitoring Cr, Cu, Mg, Mn, and Ni presence in the melt. Besides, Kondo et al. [8] reported a little difference in plasma temperatures between solid and molten steels, narrower linewidths for molten steel than solid steel plasmas, the electron density of molten steel was 46 % of that for solid steel and the atomic density in plasma for molten metals was 1.3 times that for solid steel.

However, LIBS applications based on performing laser ablations on the top of a liquid present drawbacks due to the lack of renewal and stability of the analyzed surface. Indeed, the bath surface exposed to the furnace atmosphere may be chemically modified (oxidized or nitrated) leading to the presence of a slag. Therefore, it is not representative of the chemical composition of the melt. Besides, at high temperatures, vapors may interfere with spectroscopy laser beam. In some cases, the spectrum emitted by the plasma can be masked by the hot metal emissions as it behaves like a black body [9].

Taking into account these difficulties, several devices have been developed to apply LIBS on molten metals, starting with immersion probes that presented difficulties in the optics aligning between two uses and their fouling [10]. These beam alignment problems were solved by connecting the laser to the probe with an optical fiber. Ray et al. [11] proposed such a device for the aluminum industry. Another intrusive measurement approach consisted in making a lateral opening through the crucible and bubbling an inert gas into it [12]. Laser ablations were then performed directly in the bubbles, on a clean gas/metal interface and renewed over time. This device application was quickly abandoned because metal tended to infiltrate equipment, besides LIBS measurements were local and were carried out at a fixed point in the bath. To avoid all lateral bubbling disadvantages, a team of the Canadian National Research Council (CNRC) filed a patent on the use of a bubbling rod slightly inclined to the vertical for melt probing [13]. Laser sampling is carried out in a molten metal core at the gas bubble bottom. As a case study, some investigations were carried out in silicon liquid in several works whose aim was to demonstrate the feasibility of such analysis and set a quantitative analysis model for boron [9,14,15]. This device allows obtaining reliable and accurate representative results. However, the reliability of the measurements requires a deep statistical analysis to overcome bubbles instabilities.

In this paper, a mechanical stirring of the melt by innovative rotary blades are proposed to be combined with LIBS. Their rotation will generate a representative, renewed, and stable surface as target of the LIBS laser for an in-situ analysis at high temperatures. First, we describe the design and manufacturing of the rotating blades. Then we present the experimental tests performed to predict the flow generated by their rotation in silicon melt chosen as the case study of a melt metal at high temperature. Finally, we report on the design of the association of the blades with the LIBS considering the thermomechanical and dimensional constraints of the furnace for the case study.

2. Experimental

The first step was to design an innovative rotary blade that allows obtaining a representative, renewed, and stable analysis surface. Different shapes were designed and realized using a 3D selective laser sintering.

2.1. Designed rotary blades: 3D conception

Taking into consideration theoretical parameters necessary for generating an efficient mixing [16] and crucibles dimensions, two types of stirring blades have been designed.

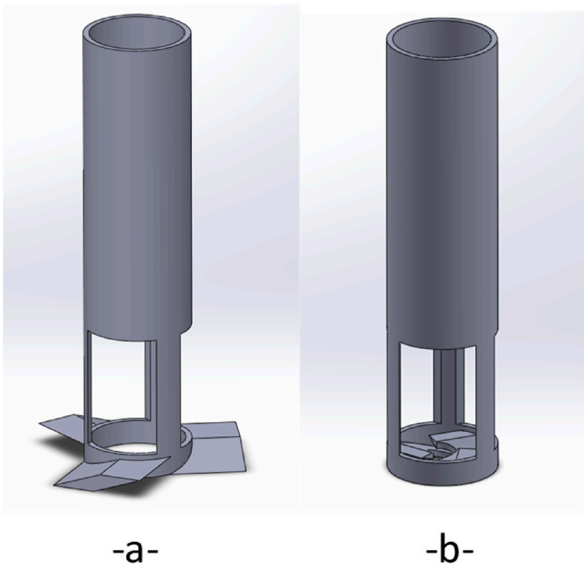

Fig. 1. Designed rotary blades with (-a-) external and (-b-) internal wings (patent FR2019052594).

One has external wings (Fig. 1a) and the other internal ones (Fig. 1b). Both of them are hollow and contain orifices above the blade in such a way that their rotation will generate a melt circulation through the orifices presented in Fig. 1. Geometrical details are given in Fig. 2.

2.2. Preliminary tests

Experimental tests at room temperature in water allowed predicting the flow generated by the designed blades rotation using a dimensionless analysis established by setting up a flow monitoring system based on particle imaging velocimetry (PIV) principle [17–22] to confirm the efficiency of mechanical stirring in obtaining a stable, representative, and renewed surface.

2.2.1. Tested liquid: water

Tests are performed in water since its flow at 20 °C is close to the selected molten metal at high temperature (silicon) as explained in Table 1 where ρ is density, μ is dynamic viscosity, Re is Reynold's number, **V** velocity and **D** diameter.

Indeed, Reynold's number that represents the relationship between the inertia forces and viscous forces is quite similar in both cases [23]. Therefore, the two fluids can, in the first approximation, be considered similar with the same geometry flows into the water. Consequently, the behavior of the surface targeted by the laser must be identical in both cases.

Testing rotary blades are manufactured in polyamide using the 3D selective laser sintering (SLS) printing technique, which, like most 3D printing processes, forms an object layer by layer from a digital file. This technique is mainly distinguished by its precision and using powder printing materials in addition to a laser for material hardening.

Plexiglas crucible, in which tests are carried out, is cylindrical and transparent. Its external diameter is 125 mm and the internal diameter is 115 mm with an external height of 240 mm and an internal height of 228 mm.

It is worth mentioning that in the case of other metals, proper rheological properties of the metal must be considered and tests in water are only valid in the case of molten silicon case and are not valid for other metals.

2.2.2. Flow visualization: tracers, lighting, and image acquisition

Water flow monitoring is achieved by using tracers. Indeed, light reflective particles (10–60 μm in diameter) covered with silver (Iriodin 100 particles) are added to water. These tracers locally follow water movement as their density is close to the water's one to avoid sedimentation. Besides, a few drops of concentrated rheoscopic fluid (Pearl Swirl) are added to better visualize the fluid movement and the current lines. It is a suspension of thin elongated platelet particles that align under the effect of velocity gradients [24].

A thin vertical plane crossing the crucible center is illuminated by a light source to prevent the overlapping of particle images located at different distances from the observer. The light source is a lighting projector to which a shutter was added so as to allow the lighting of a vertical plan crossing the crucible.

An acquisition system (Panasonic DMC-FZ18 ISO 200) allowing liquid flow recording is placed in front of the transparent crucible and perpendicular to the illuminated plane as presented in Fig. 3. The resolution of the camera is 72 dpi × 72 dpi and 2560 pixels × 1920 pixels.

2.3. Validation tests on molten metals: silicon melt as a case study

The complete probe was designed and realized considering the thermomechanical [25–28] and dimensional constraints of the

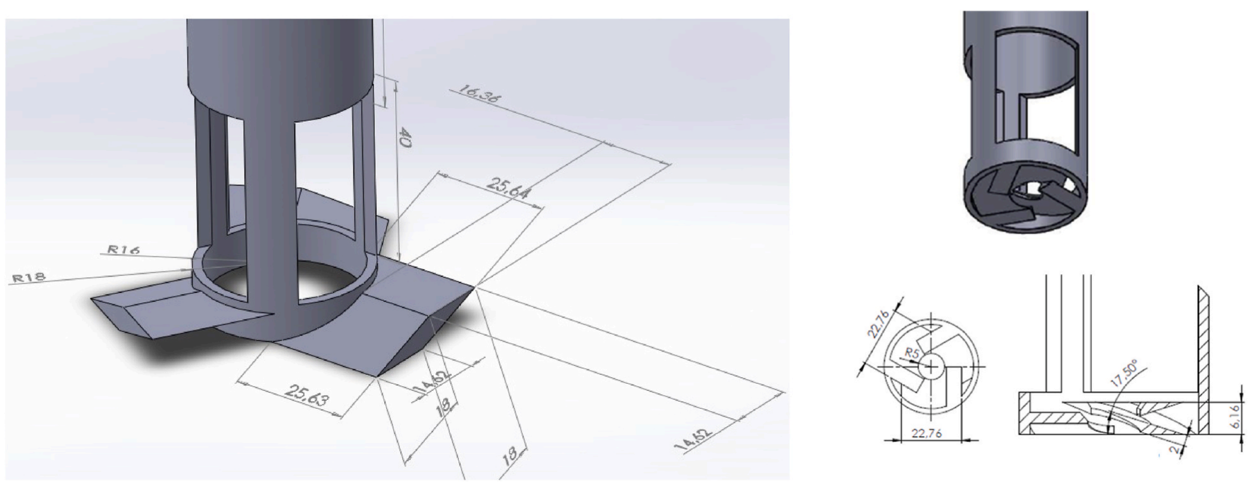

Fig. 2. Geometrical details of the designed blades with the attack angle.

Table 1
Physical properties of water at 20 °C and silicon melt at 1414 °C [23].

	Water (20 °C)	Silicon melt (1414 °C)
ρ (kg·m^{-3})	1000	2550
μ (Pa·s)	10^{-3}	7.5×10^{-4}
Re $\left(\frac{\rho VD}{\mu}\right)$	1376	1495

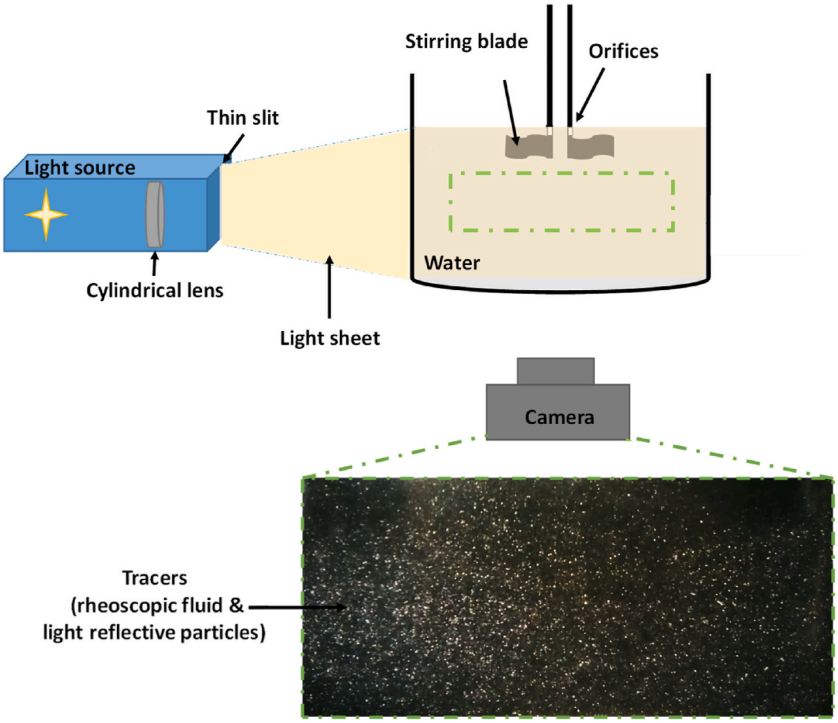

Fig. 3. Experimental flow monitoring generated by the rotary blades stirring.

Fig. 4. Graphite blade connected to alumina hollow rod.

furnace and used in molten metal at high temperature (silicon). This design can be adapted to all kinds of molten metals whose compositions need to be monitored in situ to reduce time and cost analysis.

To validate the designed rotary blades at high temperatures, testing blades were manufactured in graphite from Mersen2020. An experimental test has been realized using 3 kg of electronic silicon (EG-Si) charge melted in a cylindrical graphite crucible (height h = 240 mm, diameter d = 124 mm) whose dimensions are quite similar to the one used for experimental tests at room temperature using water. The melt was generated in an electrical resistance furnace (~1450 °C).

The blade was connected to the alumina hollow rod through which the laser passes (Figs. 4 and 5) using the paste “Adhesive V58a” manufactured by the SGL Carbon Group. The paste is based on synthetic resin with graphite as filling material. Since the glue will be subjected to temperatures above 180 °C, it is necessary to heat it under a vacuum at a heating rate of about 10–20 °C/h up to 600 °C. The external diameter of the alumina rod is 18 mm and the internal one is 14 mm.

To permit the introduction of the blade inside of the molten metal in the furnace, a system that controls the hollow alumina rod rotation (hollow rotating passage) and its vertical translation (bellows) were designed (Fig. 5). These elements were made from stainless steel as they are not submitted to high temperatures. The internal diameter of the hollow rotating passage is 22 mm. It is linked to an eccentric stepper motor (NEMA23 XL) by a belt (Fig. 5). The software monitoring the rotation is PhidgetStepper Bipolar HC. In order to avoid the rotation of the LIBS while the blades and alumina rod are rotating and to avoid the use of a glass window which is likely to be changed regularly, the connection between the upper flange of the rotating passage and the LIBS has been designed in such a way to generate the rotation without the rotation of the flanges. In the case of vapors presence, the LIBS machine can introduce argon gas through the alumina rod. The experiment presented in this paper was realized under a vacuum environment. The probe was designed in such a way as to ensure that the air would not be introduced into the molten metal.

3. Results

3.1. Description of water flow induced by the mechanical stirring

In the case of the stirring blade with internal wings (Fig. 6a and Video 1), the contact between water particles and the leading edge corresponding to the lower part of the wing will induce particles upward causing a vacuum on the other side of the wing. This will induce vertical water displacement from the blade bottom of the free surface which will be targeted by a LIBS laser beam of LIBS (suction movement). Besides, orifices’ presence allows a horizontal evacuation of particles that reached the free surface and so join the other particles moving with axial flow.

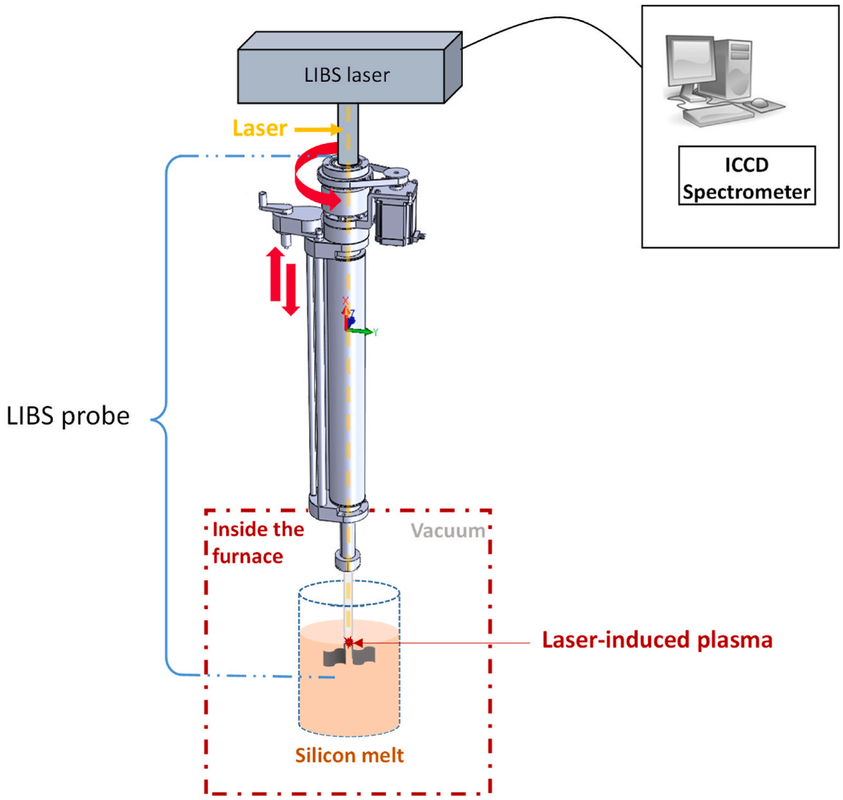

Fig. 5. Schematic of the immersed LIBS probe.

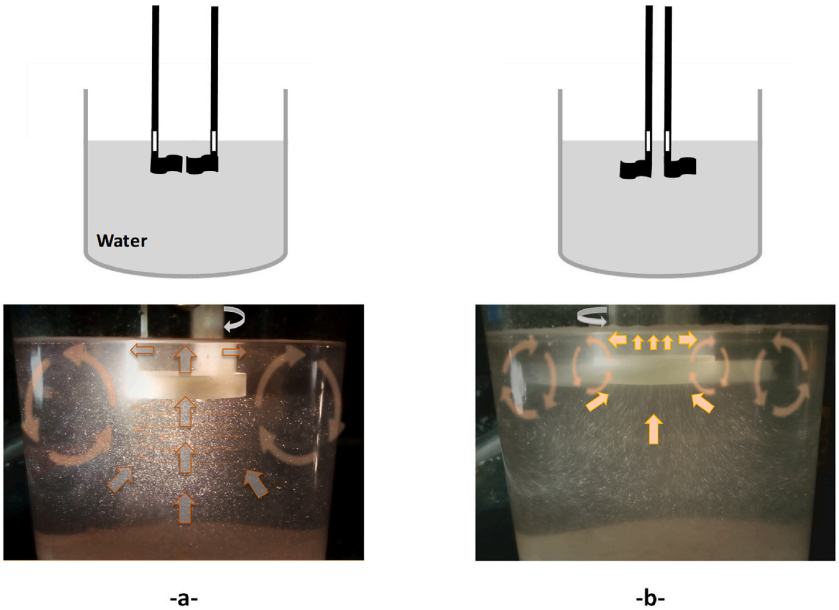

Fig. 6. Water flow generated by mechanical stirring using the two designed blades: external (-a-) and internal wings (-b-) (from Videos 1 and 2).

As presented in Fig. 6b and Video 2, using a stirring blade with external wings, a significant suction movement takes place, which induces a rise of a large number of particles upwards via the blade central diameter. These particles reach the water-free surface, then, similarly to the other blade; they are evacuated through the orifices intended for this purpose. At the wing tips, water particles that are in contact with the leading edge tend to slide up or down on the wing depending on the stirring direction.

3.2. Influence of stirring velocity

Different stirring velocities have been tested using the two stirring blades. The behavior noticed for the blade with internal wings is the following:

- *at 25 rpm*: The suction movement, particle horizontal evacuation, and loops that characterize axial flow are present. After 20 s, suction movement is carried out in a helical way as shown in Fig. 6a due to the slight entire water rotation influence.
- *at 50 rpm*: Suction movement intensity is higher and the particles rate reaching the free surface is greater. Water flow is the same except that the disturbance, due to the overall water movement, is accentuated with a slight vortexes' formation.
- *at 75, 100 rpm*: From the beginning, the suction area is wider and particles reaching the free surface rate is higher. The movement in axial flow is more pronounced. From the 15th second for 75 rpm (13th for 100 rpm), the movement is strongly disrupted by the overall rotation and there is vortexes formation in the crucible center. Thus, the particles can no longer reach the free surface because they find themselves making a circular movement to follow the overall movement.

Quite similar behavior has been noticed for the blade with external wings in terms of the presence of suction movement and its disruption due to the overall rotation of the liquid. The stirring duration is disturbed after 20, 15, 9, and 8 s for a rotation velocity respectively, of 25, 50, 75, and 100 rpm.

Loops' behavior and suction movement intensity depend on stirring direction. Indeed, during a clockwise (or counter-clockwise) direction, at the wing tips, particles that are in contact with the leading edge tend to rise (slide down) on the wing, which generates an axial loop movement at the wing ends.

It is worth mentioning that the overall water tangential movement, which disrupts suction movement and is behind the vortex generation, is due to the crucible cylindrical shape [29,30]. To prevent this movement occurrence, baffles may be used to transform a part of the primary tangential rotational movement into a mainly axial tri-directional movement. These baffles extend beyond the free surface of the liquid and extend to the crucible bottom. For a square crucible, it is not necessary to add baffles because the angles play this role. As it is not always possible to guarantee baffles' presence in industrial applications used for silicon purification, stirring parameters have to be optimized to avoid the presence of the liquid overall movement and any source of disturbance such as vortexes.

3.3. Visualization of the stable, representative, and renewed analyzed surface by LIBS

To better visualize the renewal of the analyzed surface that will be targeted by the LIBS laser during stirring, a significant amount of

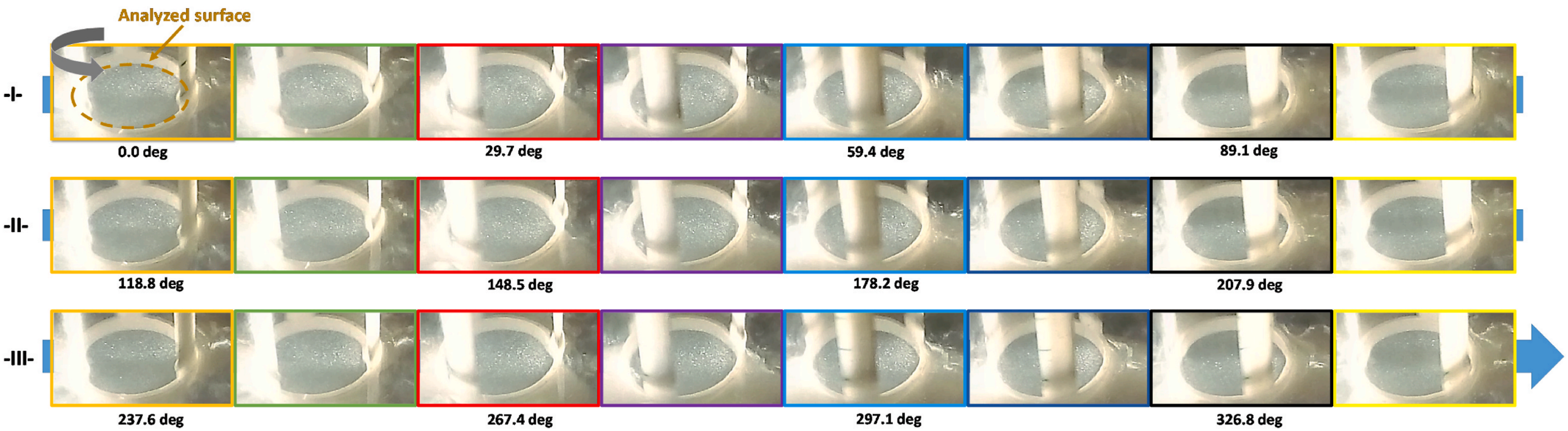

Fig. 7. Frames recorded during the blade rotation in the water.

rheoscopic fluid was introduced into the water.

Fig. 7 represents some frames recorded during one rotation. This latter included 72 frames. In this figure, one in three frames has been selected. Each line (-I-, -II, -III-) corresponds to one-third of a rotation. Frames with the same outline colors correspond to quite a similar view of the analyzed surface with the same lighting conditions which can be influenced by the position of the blade sides. White points which are present on the surface of the liquid and localized in the central part of the blade (analyzed surface) correspond to rheoscopic fluid combined with the reflective particles. The stirring velocity was 25 rpm.

The comparison is made by observing the distribution of reflecting particles (white points) between two successive images corresponding to a rotation of approximately 15°. It is evident from Fig. 5 that the distribution of these reflecting particles changes noticeably between the two successive images, indicating that the surface of the analyzed sample is undergoing changes as the blade rotates thanks to the orifices' presence and mechanical stirring.

Besides, in this figure, the analyzed surface is stable due to vortexes' absence or any turbulence zone. This is guaranteed by the laminar flow (Re = 1277) through the hollow internal diameter.

Furthermore, with this visualization configuration, it is also possible to underline the water current lines' evolution, which are present in Fig. 8. This validates the representativity of the analysis surface dedicated for LIBS measurements.

After a continuous stirring in the same conditions, surface renewal is still taking place. However, the vertical movement of particles manifests in the form of helicoidal movement as shown in Fig. 9 due to crucible geometry and baffles' absence. Fig. 9 complements the understanding of the impact of blade rotation on the analyzed surface. It presents frames (Fig. 9a–f) corresponding to two successive rotations, with each frame representing one-third of the complete rotation. In each frame, the analyzed surface is distinctly different from the others. This highlights the dynamic nature of the changes occurring on the surface due to the rotation of the blade. These frames provide a clear visual demonstration of how our technology results in a renewed and varied analyzed surface, which is crucial for achieving stable and representative LIBS results.

3.4. Validation on molten metal: silicon melt

3.4.1. Generation of representative, stable, and renewed surface

The two blades allowed the obtention of renewal, representative and stable of the surface analysis at room temperature. The central suction flow and the mixed areas in the liquid in the case of the external wings blade are higher than the internal wings blade. For this reason, the blade with external wings was selected for high temperature validation thanks to its efficiency in terms of renewal and representativity. Besides, graphite machining of the blade with external wings is easily feasible in comparison with the internal wings blade.

The blade was lowered by the designed system localized outside of the furnace (Fig. 5) to above the crucible; it was held in this position so that it could heat to avoid any sudden thermal shock. Since graphite is a good conductor, the length of time the blade was held in this position was short (10 min). Then, it was introduced into the crucible and held for 10 min in it before reaching the silicon bath. After reaching the surface of the silicon bath, different positions were identified to perform the stirring tests at different velocities. The optimized rotation speed adopted in molten silicon is 7 rpm. The direction of stirring used is the one defined before (counter-clockwise).

The oxides, generated from a reaction between melt and residual oxygen, were directed outwards by the blade rotation and then carried away by silicon flow (suction movement and axial loops) through the orifices (Fig. 10). The continuous rotation of the blade thus prevents the reintroduction of oxides into its center, and so, guaranty renewable and representability of the surface that will be targeted by the LIBS laser.

It is worth mentioning that in the presence of air, stirring will accelerate the oxidation and so the chemical composition modification. Besides, it will allow at the same time the generation of a renewal analyzed surface which can be targeted by the laser to analyze the evolution of the bath composition (as shown in the case of oxides generated from residual oxygen in the furnace in Fig. 10 and in

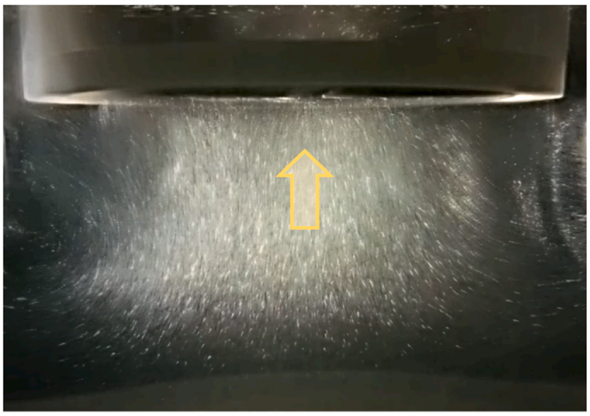

Fig. 8. Visualization of the current lines generated by stirring.

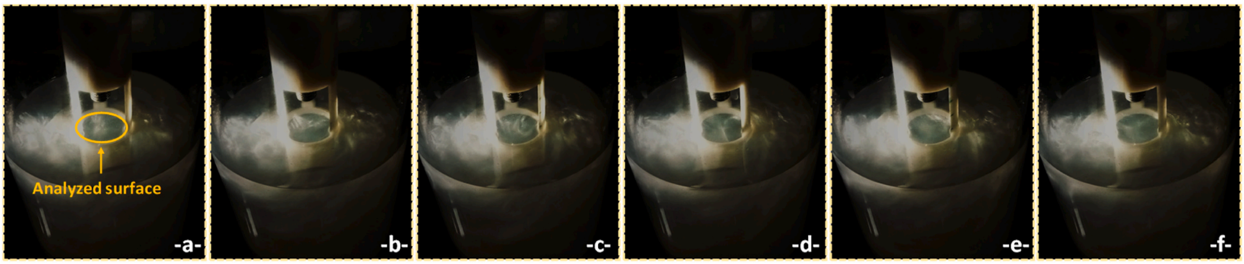

Fig. 9. The analyzed surface renewal during two successive rotations at room temperature.

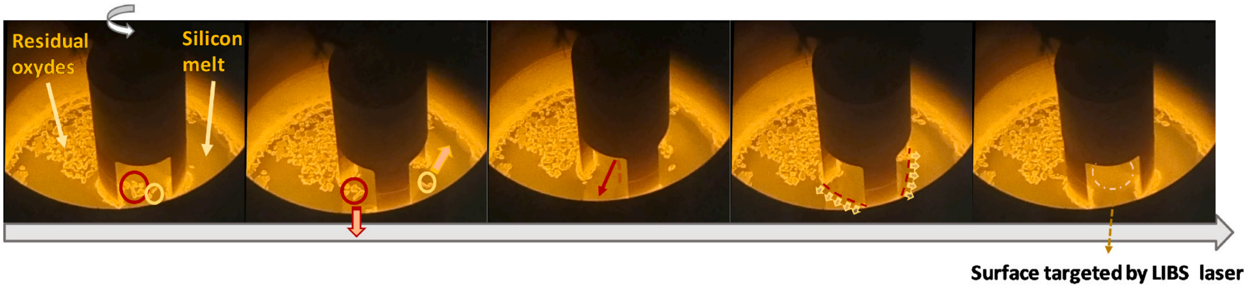

Fig. 10. Generation of a stable, renewable, and representative surface targeted by LIBS pulse laser at high temperature (from Video 4).

Video 4).

The surface stability has been obtained using an optimized stirring velocity. Its value has been calculated from the velocity of experimental tests realized in water by considering the similarity between flow generated in water (Re_{water}) and the flow generated in silicon melt (Re_{Si}) using the following equations:

$$\mathrm{Re}_X = \frac{\rho_X V_X D}{\mu_X} \tag{1}$$

where X = Si, water

Re: Reynolds number, V: velocity ($m{\cdot}s^{-1}$), D: diameter (m), ρ: density ($kg{\cdot}m^{-3}$), μ: dynamic viscosity ($kg{\cdot}m^{-1}{\cdot}s^{-1}$).

From (1), Re_{Si} can be written as follow:

$$\mathrm{Re}_{Si} = 3,4.\frac{V_{Si}}{V_{water}}.\mathrm{Re}_{water} \tag{2}$$

For a similar flow $\mathrm{Re}_{Si} \sim \mathbf{Re}_{water}$, then:

$$\omega_{Si} = \frac{\omega_{water}}{3.4} \tag{3}$$

The flow generated by stirring with the outer wings blade guaranteed the renewal of the analytical surface, its representativeness, and stability.

3.5. Generalization for other spectroscopic techniques

Mechanical stirring developed in this work and validated in molten silicon allowed obtaining a renewed, representative, and stable analyzed surface dedicated for spectroscopic measurements using LIBS as an example represented in Video 3. The designed probe presented here can be, then, applied for other spectroscopic techniques requiring in situ reproducible and representative measurements in liquids in hostile industrial environments.

Among these techniques, mass spectrometry techniques which are based on identifying elements thanks to a local ionization and subsequent mass analysis of the generated ions can be quoted. Compared to LIBS, these techniques may afford a better measurements sensitivity, however, they present some difficulties in the transportation of ions to the sensor. These problems have been described by Johnson et al. [31] who proposed the use of an ion funnel for in situ sampling.

Among mass spectrometry techniques, laser ionization mass spectrometer (LIMS) [32–34] can be coupled with the presented probe here. LIMS operates by removing ions from the sample using a micro-focused high-density laser beam, and then analyzing the mass of these ions. The resulting ions generated by this laser are then analyzed with Time-of-Flight (ToF) mass spectrometry, wherein the mass values of the ions are quantified based on their transit time across a high vacuum region through which they are accelerated by an electric field of known value. It allows having the composition, concentration, and in the case of organic molecules structural information. The main advantages offered by LIMS compared to LIBS are its high elemental sensitivity, wide mass analysis range, and relatively small beam diameter (1–5 μm).

4. Conclusion

This paper proposed an innovative engineering approach for in situ analysis of molten silicon using spectroscopic techniques and especially using Laser induced breakdown spectroscopy. This latter has serious limitations in the case of molten metals due to slag formation, masking radiation emission lines, or laser jamming by vapors. Different devices were developed to overcome these difficulties; however, improvements were still required. For this reason, this paper presented a solution based on the development of an easily implemented device with a rotary stirring blade to ensure a stable, representative, and renewed analyzed surface targeted by LIBS laser.

Two types of rotary blades were designed considering several key parameters such as the generated liquid flow, container geometry, and the stirring position. They were realized thanks to 3D selective laser sintering and tested using an experimental approach based on particle image velocimetry at room temperature in water. The behavior of the liquid flow generated by their rotation and the optimized stirring parameters were described in this paper. The two blades allowed the obtention of renewal, representative and stable of the surface analysis at room temperature. The central suction flow and the mixed areas in the liquid in the case of the external wings blade are higher than the internal wings blade. For this reason, the blade with external wings was selected for high-temperature validation in molten metals thanks to its efficiency in terms of renewal and representativity. Besides, graphite machining of the blade with external wings is easily feasible in comparison with the internal wings blade. The mechanical stirring using the designed probe applied at high temperatures in molten metals with optimized stirring parameters allows obtaining stable, representative, and renewed measurements surface dedicated for different spectroscopic measurements in hostile environment.

CRediT authorship contribution statement

Y. Belrhiti: Writing – review & editing, Writing – original draft, Visualization, Validation, Investigation, Formal analysis, Data curation, Conceptualization. **M. Albaric:** Methodology, Conceptualization. **M. Benmansour:** Supervision, Methodology, Funding acquisition. **J.-B. Sirven:** Writing – review & editing, Supervision, Funding acquisition. **A. Chabli:** Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:Younes Belrhiti has patent licensed to FR2019052594 and US11867632B2.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.heliyon.2024.e25626.

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