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Ester-Based Lubricant and Anti-Leidenfrost Additive Solutions on Aluminum High-Pressure Die-Casting Applications

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Abstract: The high-pressure die-casting process is growing since it is a cost-effective solution in the production of lightweight parts for a variety of industries. Nevertheless, the harsh working conditions of the die lead to premature failing and poor quality of the produced parts. Lubricants are applied to cooling the die surface and create a protective film to minimize die wear. However, the high temperature of the die during the casting production makes it difficult for the lubricant to reach the die surface due to the Leidenfrost effect. In this study, the effectiveness of newly developed ester-based lubricants designed to address Leidenfrost phenomenon in high-pressure die-casting is evaluated at laboratory and pilot plant scale. The new lubricants are based on the same ester solution; however, one of them includes a specially formulated anti-Leidenfrost additive to optimize performance at the temperature ranges typically encountered in industrial aluminum high-pressure die-casting processes. The results show a correlation between lubricant heat-transfer capability and aluminum adhesion. Additionally, a pilot plant methodology for testing newly formulated lubricants has been established while the experimental methodology developed for assessing heat-transfer capability is validated as a rapid and cost-effective approach for evaluating lubrication alternatives for high-pressure die-casting applications. Finally, the efficiency of environmentally friendly ester-based lubricants for high-temperature applications has been demonstrated.

Keywords: adhesion wear mechanism; Leidenfrost point; aluminum high-pressure die-casting; additive lubricants

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1. Introduction

The high-pressure die-casting (HPDC) process is growing since it is a cost-effective solution in the production of lightweight parts for a variety of industries including automotive, electronics, aerospace, consumer goods and industrial machinery industry [1–4]. HPDC enables the production of complex mechanical parts with high dimensional accuracy and smooth surface finishing, also enabling the production of thin-walled parts at high production rates [4,5]. Low melting alloys such as aluminum, zinc, magnesium or copper alloys are widely used in HPDC industries [6]. Aluminum is the most used non-ferrous metal, playing a crucial role in various applications within the automotive and aerospace industries [1]. For instance, when utilized in vehicles, aluminum can reduce weight by much as 50% compared to conventional mild steels structures [1]. Moreover, aluminum exhibits a good corrosion resistance, specific strength, high stiffness and excellent fatigue resistance [6–9]. Nowadays, innovative aluminum alloys and advances in casting technologies are being developed to enhance strength among other properties. As a result, the casting of larger and more complex components is now achievable [10]. In aluminum HPDC process, aluminum alloys are injected into a steel die at high pressures (7–140 MPa) [2,3] to guarantee that the metal fills every detail of the mold. Typically, the die is made of hardened steel [11,12], the surface of the dies must be cleaned and lubricated [2] to ensure an optimal removal of the part. Once the casting has fully solidified and sufficiently cooled down, the die is opened, and the part is ejected using activated pins [2,5]. Although HPDC offers numerous advantages, achieving high-quality parts heavily depends on the steel die, which accounts for approximately 20% of the total production cost [12]. During HPDC process, the die experiences extreme conditions due to a combination of mechanical and thermal loads [12], with surface temperatures reaching up to 450 °C [5,13,14]. The steel die undergoes severe thermal shock and impact loads from the injection of molten alloy at high pressures. These harsh working conditions lead to die failure primarily due to soldering, thermal cracking and washout [6,12,13,15].

The production of structural castings and other parts with relevant mechanical requirements, first introduced in the 1990s, has experienced remarkable growth in the automotive industry over the past decades and shows strong future potential [15]. This innovation has transformed the sector by enabling the production of large, thin-walled parts in a single casting process. This advancement significantly reduces the number of components required to manufacture the body-in-white (BIW) structure of a vehicle, optimizing production and enhancing efficiency [16]. The high ductility required for these components is normally achieved by using primary alloys with a reduced Fe content [16–18]. However, this low Fe content significantly increases the affinity of the aluminum alloy for the steel of the die, decreasing the die's lifespan [17,19]. Consequently, the precise application of a release agent becomes crucial to minimize interaction between aluminum and steel, thereby protecting the die and maintaining production efficiency.

To prevent premature failure of the HDPC dies, the die surface is sprayed with a release agent after the part solidifies and the die is opened [18]. This step is performed before closing the die again for the next injection of molten alloy, delivered at high temperature, speed and pressure. Release agents in HPDC serve two primary purposes: (1) cooling the die surface, especially in regions where the internal cooling channels are insufficient and (2) protecting the die with a lubrication film acting as a release agent that prevents damaging mechanisms, such as die soldering or oxidation, and enabling the extraction of the casted parts [5,18]. However, the high temperatures of the die during the casting production, make it challenging for the lubricant to effectively reach the die surface due to the Leidenfrost effect. This phenomenon occurs when liquid droplets are sprayed onto hot die surface, forming a vapor layer caused by rapid evaporation. The vapor layer prevents the lubricant from making direct contact with the surface, hindering its ability to uniformly coat the die [8,20,21]. It has been reported that surface temperature and liquid saturation temperature determine the film evaporation, boiling nucleation, boiling transition and boiling film regimes [21,22]. When lubricant droplets impact the surface, the Leidenfrost phenomenon occurs because their boiling point is lower than the die's surface temperature. Consequently, to improve the efficiency of lubricants in HPDC process, newly formulated lubricants should aim to maximize the Leidenfrost point. This approach allows the lubricant droplets to contact the die surface, effectively cooling it and reducing die wear mechanisms.

The literature shows that Leidenfrost temperature point (LFP) depends on the surface roughness [20,22–24], steel surface material [23,24], droplet size [22–24] and droplet deposition method [18,22,24]. One approach to mitigating the Leidenfrost phenomenon is the development of high-performance lubricants, enabling the lubricant drops to wet the die surface at higher temperatures. This allows a lubricant film to form earlier, reducing lubricant consumption, enhancing die surface protection, and ultimately extending the die's service life. The efficiency of new formulated lubricants relies on its heat-transfer capacity, which depends on the contact area between the lubricant droplets and the die surface [18]. In that sense, the wettability of the lubricant at high temperatures is enhanced through the use of additives. For instance, the presence of ionic surfactants is proved to reduce the LFP on different substrates [22]. The evaporation of lubricant droplets is significantly influenced by both contact angle (CA) and contact radius; they evaporate more quickly when the CA and the contact radius are low. Nevertheless, the relationship between the CA and LFP is not straightforward; therefore, it is not possible to optimize both through lubricant formulation. Higher LFP values correspond to higher CA values, which results in poorer wettability [23]. The literature shows that a hydrophilic surface with a CA $< 90^\circ$ enhances the LFP. Moreover, a critical contact angle exists, allowing the prediction of the Leidenfrost phenomenon occurrence based on the initial surface temperature [24]. The influence of spray lubrication and lubricant composition on technological parameters is nonlinear [23], making it challenging to optimize lubricants based on extrapolated literature data. This complexity arises because the CA is strongly affected by factors such as temperature, surface roughness, steel type and spraying conditions.

In this study, newly formulated lubricants have been developed specially for the aluminum high-pressure die-casting industry. These novel lubricants are bio-based and environmentally friendly, using ester-based oils instead of mineral oils. While bio-based oils are known for their lower oxidative and thermal stability [25–27], these issues can be mitigated through chemical modifications and the use of proper additives [27]. Ester-based oils are known for their superior lubricity, especially in a boundary lubrication regime, low-friction coefficients and reduced wear, primarily due to their long hydrocarbon chains, which minimize asperity contact and enhance the protective surface layer [26]. Ester-based lubricants currently face several challenges that limit their widespread application across various industries. From a technological perspective, developing these lubricants is complex and requires novel chemical formulations and additives. However, bio-based lubricants are gaining significant attention due to their good lubricity and environmentally friendly non-toxic properties. These benefits make ester-based lubricant solutions attractive in several industries, and demand for such products is expected to increase in the future.

The main purpose of this work is to assess the effectiveness of ester-based lubricants and anti-Leidenfrost additives in HPDC industry by validating newly methodologies designed that meet industrial requirements. This work focuses on evaluating the effectiveness of newly developed ester-based lubricants designed to address the Leidenfrost phenomenon in high-pressure aluminum die-casting. Two lubricants are evaluated, both based on the same ester-oil solution, with one incorporating a specially formulated anti-Leidenfrost additive to optimize performance at the temperature ranges typically encountered in industrial aluminum HPDC processes. Two different approaches have been conducted to prove the effectiveness of lubricant additives to reduce aluminum adhesion on the die surface: laboratory tests and pilot plant validation. Laboratory tests aim to study the relationship between aluminum adhesion and the heat-transfer capability of the lubricants, with results compared to a commercial mineral-oil-based reference lubricant. Adhesion tests, using a ball-on-disc configuration, have been performed to measure the force required to break the adhesion bond between a hardened steel surface and an aluminum ball in the presence of lubricant. Additionally, the cooling rate during lubricant spraying was recorded and fitted to a sigmoidal model to compute the lubricant's heat-transfer capacity. The contact angle at room temperature was also analyzed and correlated with both the adhesion force and the heat-transfer capability of each of the analyzed lubricants. Pilot plant validation involved two approaches: a short series to evaluate the heat-transfer capability of the lubricants, serving as a screening test in industrial conditions, and the production of 1200 aluminum casting parts, with a die surface inspection performed after 900 parts were produced. An AlSi10MnMg alloy, widely used for manufacturing structural HPDC components, has been used, replicating standard industrial conditions. This work shows a correlation between heat-transfer capability (linked with the LFP) and aluminum adhesion, while the CA does not directly influence lubricant performance. Additionally, a pilot plant methodology for testing newly formulated lubricants have been established. Finally, the efficiency of ester-based lubricants for high-temperature applications, such as HPDC, has been demonstrated.

2. Materials and Methods

2.1. Lubricants

Two different ester-based lubricants in the form of emulsions were specially formulated by Brugarolas using a new formulated ester by Industrial Quimica Lasem (IQL). The first lubricant, an ester-based lubricants (lubricant 1), was selected to assess its effectiveness at the HPDC temperatures ranges compared to a PAO-based commercial lubricant (reference lubricant). The second lubricant, with the same ester-oil solution but a newly formulated anti-Leidenfrost additive (lubricant 2), was selected to evaluate the effectiveness of this new additive. By comparing these two lubricants, which have slight differences, it is sought to determine the effectiveness of each new formulation while validating laboratory and pilot plant approaches. Both esters and emulsions were formulated according to the requirements needed in aluminum HPDC applications. Lubricants were specially formulated to improve the demolding of the parts produced by HPDC process while promoting sustainability through the use of esters as a base oil. Additionally, a new additive was developed by Brugarolas to decrease the Leidenfrost point, allowing it to reach the die surface more effectively, thereby preventing soldering and improving the release of the part. The tribological behavior of the lubricants was tested and compared with a commercial lubricant and with the dry condition. In Table 1 are shown the description and the nomenclature of the different solutions tested.

Table 1. Nomenclature and description of the lubricant solutions studied.

Lubrication	Nomenclature	Description
Dry	D	-
Reference lubricant	R	PAO-based commercial lubricant
Lubricant 1	LUB 1	Ester-based lubricant
Lubricant 2	LUB 2	Lubricant 1 + Leidenfrost reduction additive

The lubricants were prepared by diluting the concentrated emulsions with distilled water. The dilution rate was 1.25:100 (1.25%) for all of the tested lubricants.

2.2. *Experimental Methology*

2.2.1. Adhesion Force

The efficiency of the lubricant in terms of soldering was computed at laboratory scale by means of a Bruker UMT Tribolab tribometer. In designing the experiment, industrial die-casting parameters were considered; these critical industrial parameters are summarized in Table 2.

Table 2. Industrial die-casting parameters.

Industrial Parameters	Value
Die temperature (lubricant application)	450 °C
Lubricant-spraying time	6 s
Spraying distance	25 cm
Die material	1.2344

To replicate the industrial process at the laboratory, test samples were pre-heated to 450 °C in a conventional furnace for 30 min. Afterwards, the samples were removed from the furnace and immediately perpendicularly sprayed for 6 s as shown in Figure 1. To monitor the temperature and record the drop during the spraying step, a type K thermocouple was welded to the surface samples being the temperature measuring rate of 1 Hz with a sensitivity of 41 µV/K (approximately ± 0.25 °C).

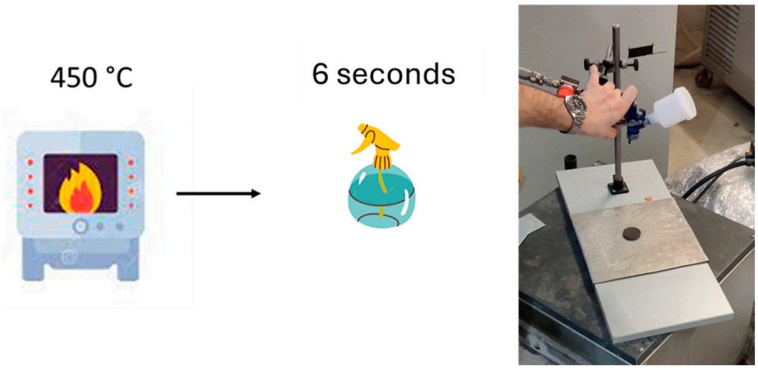


Figure 1. Lubricant-spraying experimental procedure.

Finally, the lubricant efficiency was tested by designing an adhesion test at high temperatures between a steel disc and an aluminum ball. Experimental parameters were chosen taking into account the industrial parameters (Table 2). In Table 3, the test parameters are summarized. Before the adhesion tests, all of the samples were polished up to 0.04 µm colloidal silicon suspension.

Table 3. Test parameters.

Test Parameters	Value
Temperature	450 °C
Lubricant-spraying time	6 s
Spraying distance	25 cm
Bore nozzle	0.8 mm
Air pressure	2×10^5 Pa
Disc material	1.2344
Ball material	Al 99%

Adhesion tests were performed in the tribometer chamber at 450 °C without a controlled atmosphere according to the test configuration used in previous studies [28]. The test consisted on a ball-on-disc configuration, using a 1.2344 steel disc (industrial die-hardened steel) as a lower specimen and an 99% aluminum ball of 4 mm of diameter as the upper specimen. After a stabilization period of 30 min in the tribometer chamber, the material was brought into contact. A normal force of 10 N was applied and maintained for 30 s. Subsequently, the aluminum ball was retracted at a velocity of 50 µm/s. Throughout the test, the force was recorded at each moment. When the contact between the disc and the ball was lost, a peak in the tensile force would be registered, while force would drop to zero if no adhesion occurred. A scheme of the test is illustrated in Figure 2. After the test, the residual imprints were characterized by means of 3D optical measurement system (ALICONA InfiniteFocus SL).

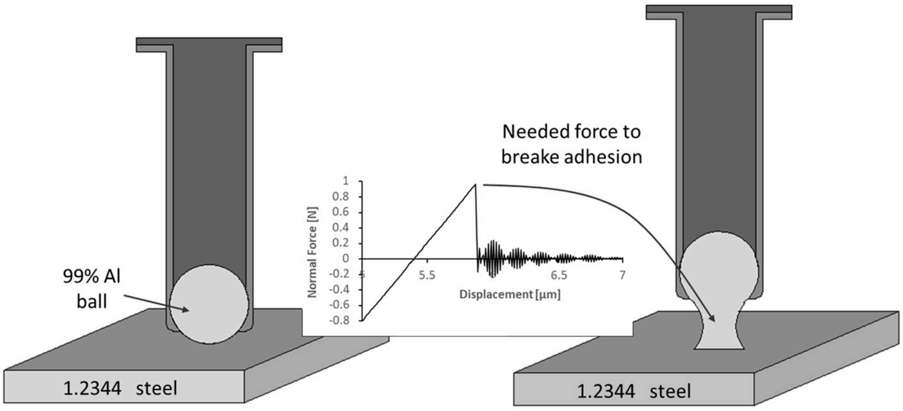


Figure 2. Adhesion test scheme.

2.2.2. Contact Angle

Contact angle (θ) measurements were performed using a Krüss DSA 100 drop shape analyzer. A total of 1.2344 steel (industrial die material) specimens were mechanically polished to mirror surface finish with a 0.04 µm colloidal silica suspension. Then, the samples were pre-heated to 450 °C in a conventional furnace for 30 min, and the contact angle between a lubricant drop and the steel were measured at room temperature. The contact angle measures the wettability of a solid by a liquid and is the angle that forms the liquid with respect to the contact surface of the solid and is determined by the adhesive and cohesive forces. In Figure 3 are shown the experimental procedure to measure the contact angle θ.

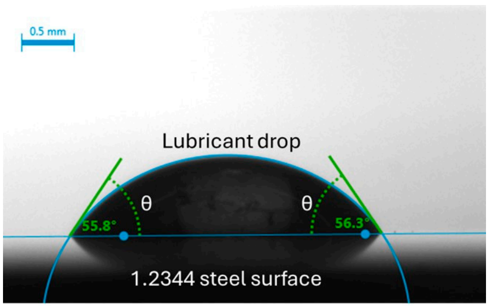


Figure 3. Contact angle θ measurement between a polished 1.2344 steel surface and a lubricant drop.

2.3. Pilot Plant Validation

To assess the effectiveness of the newly formulated lubricants in an industrial environment, several trials were performed at the HPDC pilot plant owned by Eurecat, located at the Cerdanyola (Spain) facilities. Pilot plant trials were designed to fulfill the industrial requirements; Figure 4 shows the process scheme followed in this investigation. For the casting trials, an AlSi10MnMg alloy was used with an approximate melt temperature of 720 °C and the standard casting parameters used to produce the part, replicating the working conditions typically used to produce structural HPDC components in the industry.

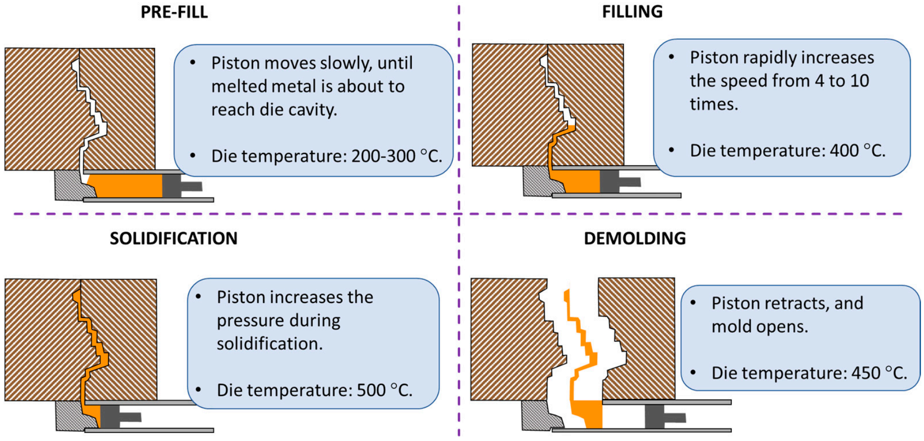


Figure 4. Pilot plant trial scheme.

The cavity inserts of the die are made of 1.2344 steel as detailed in Table 2. The die is equipped with three sensors within the cavity: two pressure sensors and one temperature sensor. The sensor placements are illustrated in Figure 5a, where the temperature sensor is centrally located (indicated by red bullet point). This temperature sensor continuously monitors the temperature value on the die surface during each injection cycle. Figure 5b displays a typical temperature curve recorded by the sensor over 10 consecutive injection cycles. When the molten aluminum reaches the die surface, the temperature suddenly rise to 500 °C before rapidly decreasing during the solidification process. The temperature sensor demonstrates excellent repeatability across the recorded cycles.

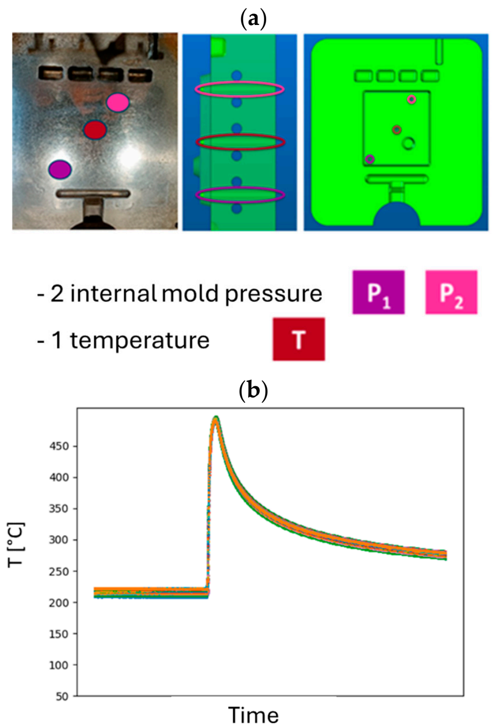


Figure 5. (**a**) Image of the die surface and the positions of the sensors; (**b**) graph scheme of the temperature evolution during 10 injection cycles (each color represents 1 injection cycle, then the repeatability is demonstrated to be excellent).

In this study, two different approaches were conducted to evaluate the effectiveness of the newly formulated lubricants:

1. Short series of 30 test parts: This analysis is aimed to assess the temperature extraction capability of the lubricants compared to the reference commercial lubricant.
2. Series of 1200 test parts: This analysis is aimed to assess the improvements in die wear mechanisms with the newly lubricant formulations. The findings can be interpretated considering the conclusions drawn from the short series.

Surface replication techniques were applied to experimentally assess aluminum adhesion during production. To ensure consistent initial conditions, the die was manually polished before using each lubricant under study. The evolution of surface roughness through the production of the 1200 aluminum parts was compared across the lubricants to track wear severity based on the applied lubricant. Surface replication techniques allow one to study the surface integrity of the original surface up to 1 µm of resolution. Surface replication was applied on areas where significant damage was observed, as illustrated in Figure 6, which provides an example of surface replication application on the surface die. The topographic characterization of the die surface was computed by means of 3D optical measurement system (ALICONA InfiniteFocus SL). Three-dimensional images of 7.5 $\times$ 0.5 mm was acquired to extract both 2D and 3D roughness parameters, facilitating a comparison of surface finishes across different wear levels. The resulting 3D images were analyzed with the SensoMap analysis software version 5.1 to determine representative roughness parameters in 2D (according to ISO 4287:Ra [29]) and 3D (according to ISO 25178: the arithmetical mean heigh Sa an the spectral parameter Sdr [30]).

(**a**) Initial state (Polished) (**b**) Damaged Die

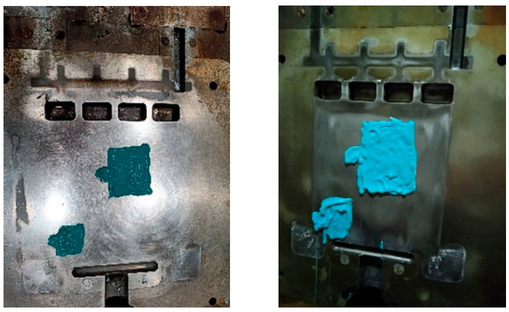


Figure 6. Surface replication technique applied (**a**) after polishing the die (initial state) and (**b**) after several die-casting cycles (damaged die).

3. Results

3.1. Laboratory Test: Lubricant Heat-Transfer Capability

Figure 7 shows the temperature curve recorded after 1.2344 steel specimens were pre-heated at 450 °C in a conventional furnace for 30 min. Then, the lubricant was sprayed for 6 s. Each point in the graph corresponds to the experimental measured points during the lubricant spraying. It can be observed that after the preheating of 450 °C, the application of the lubricant leads to a decrease in steel surface temperature. However, following the 6 s of spraying, the temperature begins to rise again due to the lack of lubricant, which is responsible for cooling the steel specimen. It can be observed that the newly formulated ester-based lubricants have a higher heat-transfer capability in comparison with the reference lubricant (mineral-oil based).

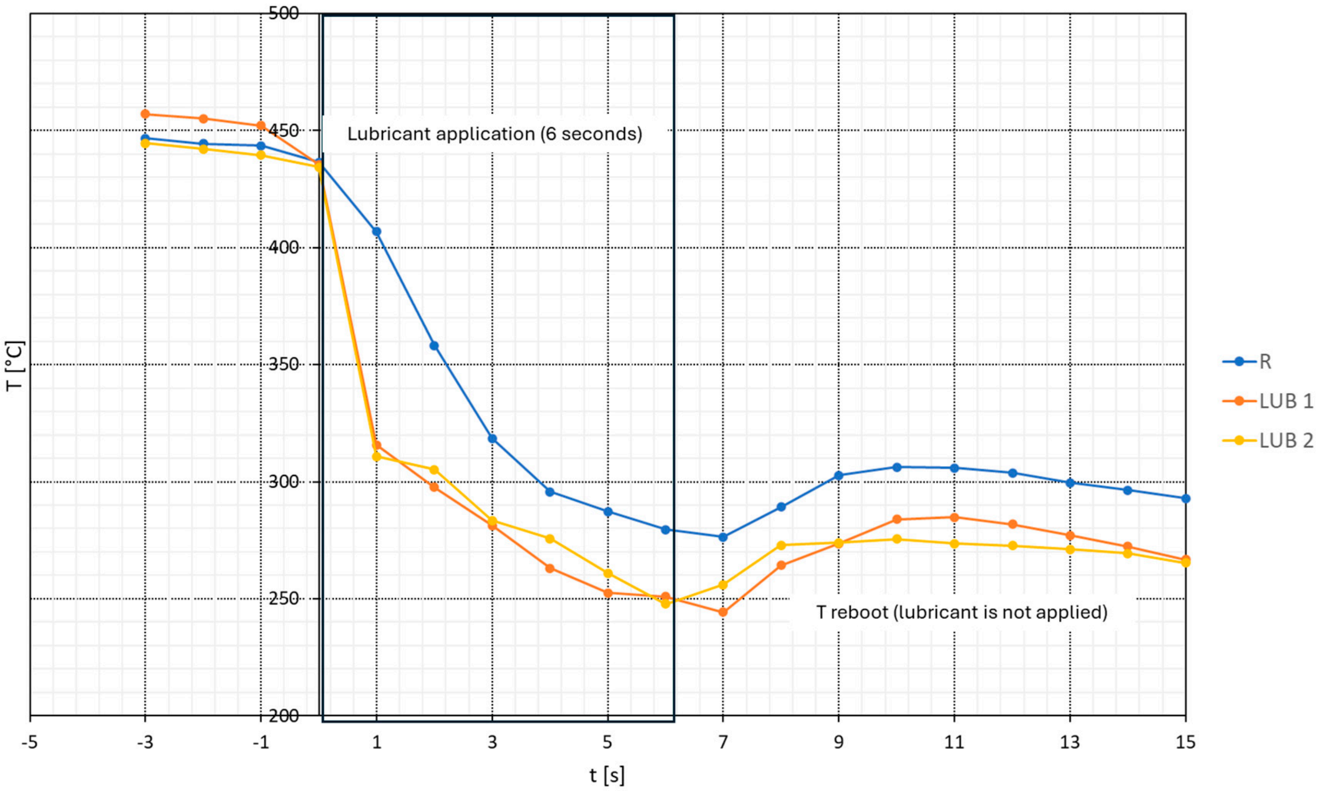


Figure 7. Monitored temperature curve for the studied lubricants.

To quantify the heat-transfer capability of the analyzed lubricants during the 6 s spraying application, the experimental points have been fitted to a sigmoidal distribution. The sigmoidal curve takes an S shape and makes it easier to understant the evolution of temperature over time during lubricant spraying. Figure 8a illustrates the temprature recorded during the 6 s lubricant spraying (dot points) by the welded thermocouples at the sample surface and following the experimental procedure described in Section 2.2.1. The dashed lines in Figure 8a illustrates the sigmoidal fitting for the recorded experimental points. The sigmoidal fitting applied to the temperature experimental data is shown in Equation (1), where A_1 is the initial temperature value, A_2 is the final temperature value, x_0 is the center and dx is the time constant.

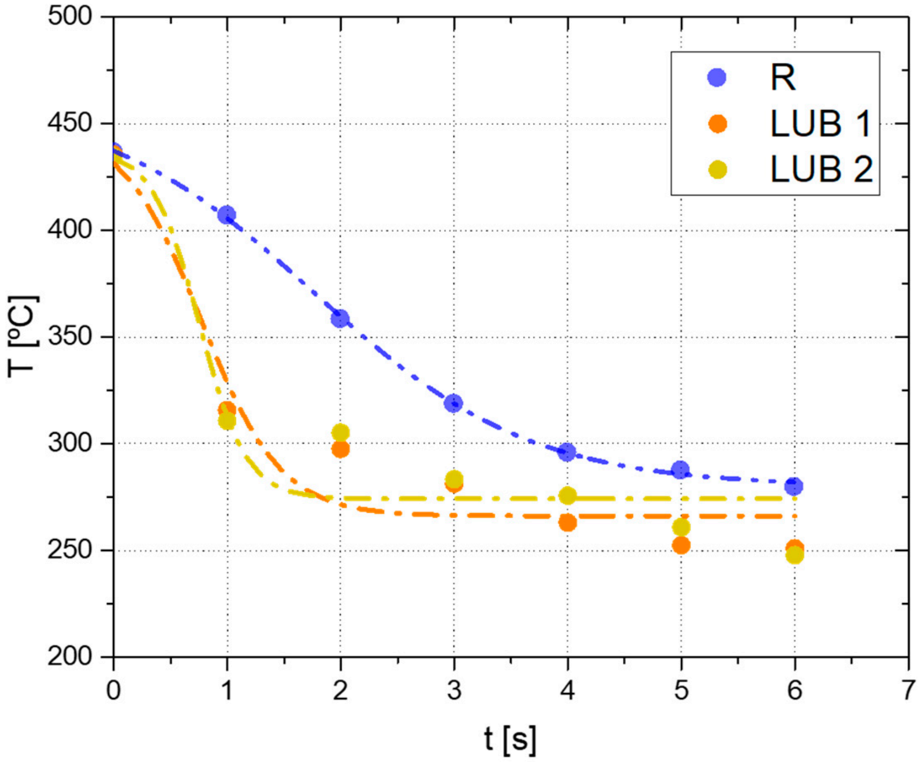
$$y = \frac{A_1 - A_2}{1 + e^{\frac{(x - x_0)}{dx}}} + A_2 \tag{1}$$

Then, the *slope* of the fitting is determined for Equation (2)

$$slope = \frac{A_1 - A_2}{4\,dx} \tag{2}$$

Using sigmoidal fitting, the heat-transfer capability of the lubricants have been assessed based on the slope of the fitted curve. Figure 8b illustrates a scheme of the heat-extraction-fitted curves, focusing on two main parameters. First, the slope of the curve (°C/s), which represents the temperature drop over time and is considered to be the heat-transfer capability of the lubricant, i.e., this value represents the Kinect of the lubricant during cooling. Second, the time at mid-slope, which represents the transition between the initial temperature (450 °C) and the stabilization temperature, is considered to be the efficiency of the lubricant (if more or less time is needed to reach the transition between the initial and the stabilization temperatures).

(**a**)



(**b**)

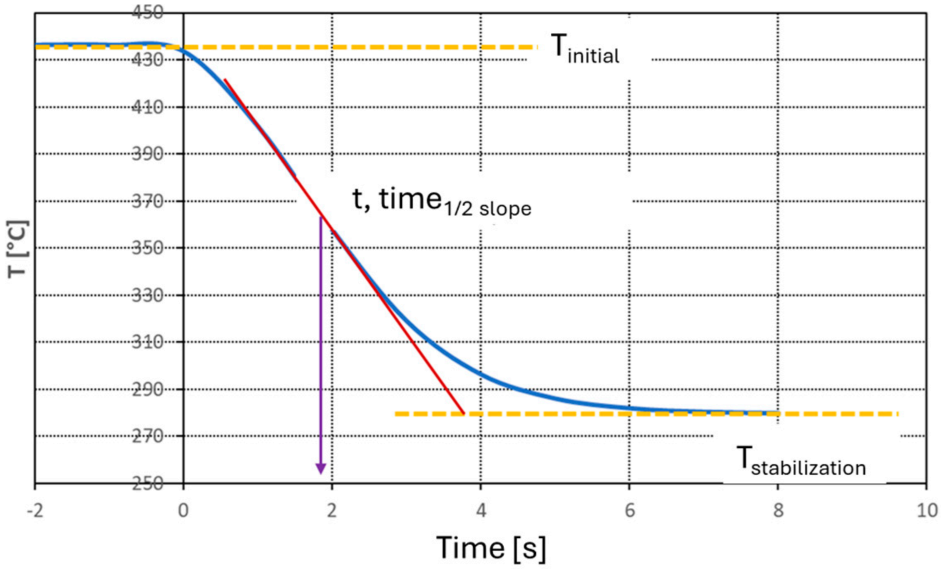


Figure 8. (**a**) Heat-extraction capacity sigmoidal fitting, where the dot points indicate the experimental temperatures recorded by the thermocouples, and the dashed lines illustrates the fitting according to the sigmoidal distribution and (**b**) Scheme of the variables evaluated with the sigmoidal fitting, where yellow line represents the initial temperature and the stabilization temperature, red line represents the slope and in the middle (purple arrow) time at mid-slope.

Table 4 illustrated the obtained results from the sigmoidal fitting in terms of the slope and the transition time. It can be observed that, despite the experimental error, the newly developed lubricant, enhanced with an additive to address the Leidenfrost point, demonstrates greater effectiveness in heat-transfer capability.

Table 4. Obtained results from the sigmoidal fitting of the experimental temperature curves.

Lubricant	$\Delta T/\Delta t$ [°C/s]	$t_{1/12}$
R	−48 ± 5.5	1.8 ± 0.1
LUB 1	−78 ± 54	0.8 ± 0.1
LUB 2	−61 ± 49	0.8 ± 0.1

3.2. Laboratory Tests: Adhesion Force and Contact Angle Evaluation

Figure 9 shows the residual imprints after the adhesion tests. It can be observed that the application of lubricants enhances the adhesion behavior in comparison with the dry condition. Moreover, the residual imprint with the newly developed lubricant, enhanced with an additive to address the Leidenfrost point, indicates a minor adhesion of aluminum at the steel specimen surface.

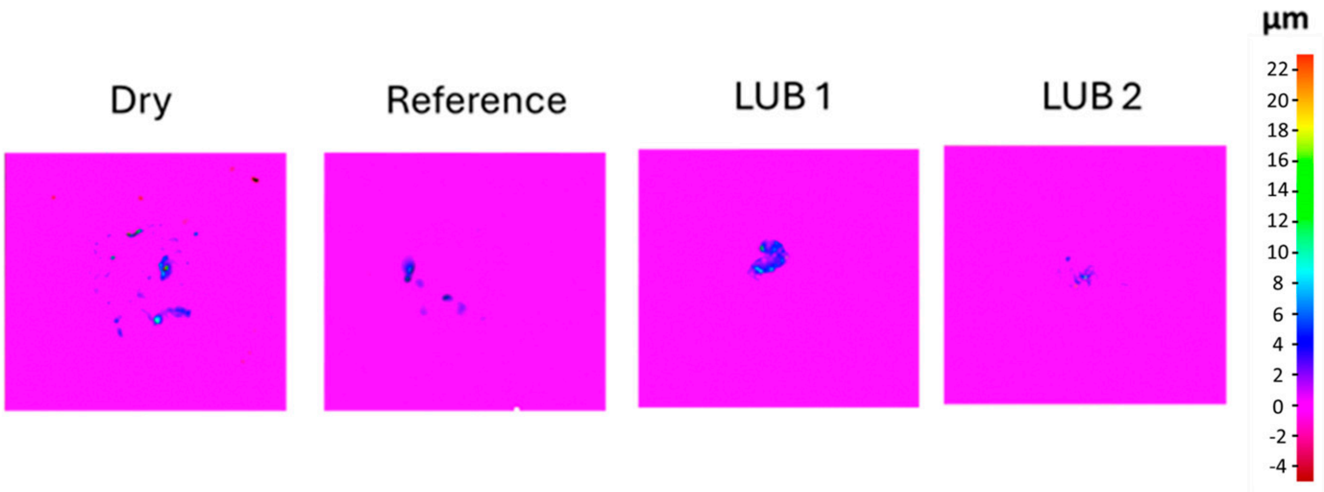
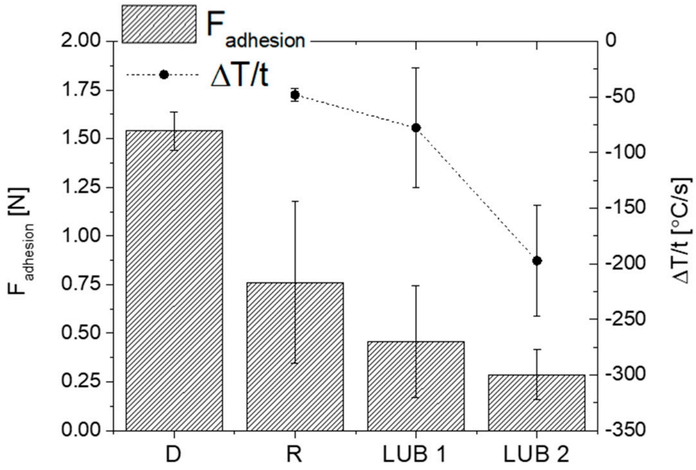


Figure 9. 3D topography images of the residual imprint after the adhesion test.

Figure 10 and Table 5 illustrates the obtained values for the adhesion force and the contact angle, evaluated as described in Sections 2.2.1 and 2.2.2, respectively. It can be observed that the adhesion force of the tested lubricants decreases with the the heat-transfer capability and the increase in the contacte angle (CA). It is worth mentioning that the CA is evaluated at room temperature, while heat-transfer capability and adhesion force are assessed at high temperature, making direct correlation difficult. Nevertheless, it has been reported that as temperature increases, the CA tends to decrease [23]. Moreover, surface roughness also affects the CA. During the pre-heating step in an uncontrolled atmosphere furnace, an oxidation layer forms on the steel surface specimen, which can influence the CA at high temperatures. This work does not address the surface effects on the CA since it is assumed that oxidation is consistent across all samples. Consequently, the results for the CA cannot be directly correlated with the adhesion force and heat-transfer capabilites computed at high temperatures.

(**a**)



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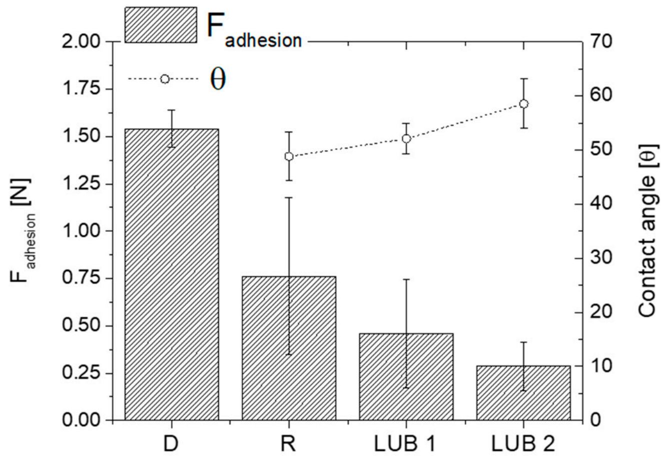

Figure 10. (**a**) Adhesion force in bars and lubricant heat-extraction capacity in dots; (**b**) adhesion force in bars and contact angle in open dots.

Table 5. Summary of the experimental values obtained for adhesion force and the contact angle.

Lubricant	Adhesion Force [N]	Contact Angle [θ]
D	1.5 ± 0.1	
R	0.8 ± 0.4	49 ± 5
LUB 1	0.5 ± 0.3	52 ± 3
LUB 2	0.3 ± 0.1	58 ± 5

3.3. Pilot Plant: Lubricant Validation

To assess the lubricant's effectiveness in an industrial environment, experiments were performed in an aluminum HPDC pilot plant. The initial step involves a visual inspection of the die to identify areas with significant wear, corrosion and material adhesion. These identified points show where replicas were applied during the pilot plant tests. Figure 11 shows the different sections into which the die surface was divided for wear inspections during the trials. Prior to each trial, the die surface was manually polished as described in Section 2.3.

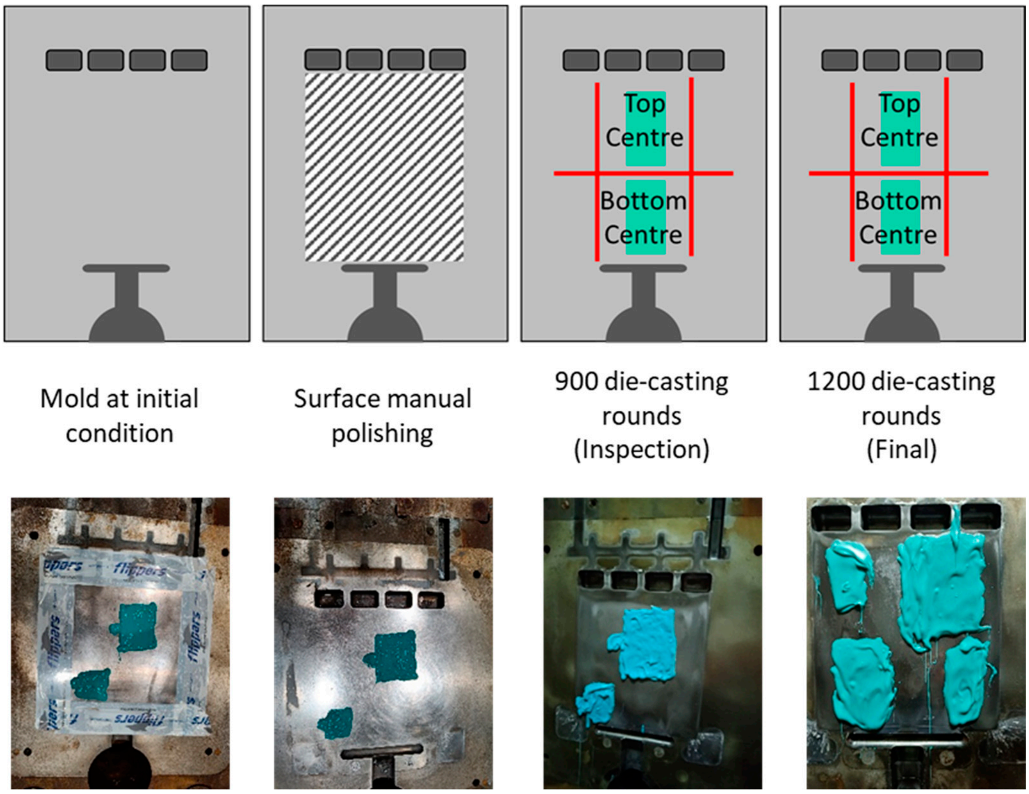

Figure 11. Scheme of the mold surface area analyzed and its division for wear inspection (**top**); images of the surface replicas applied during the inspections (**bottom**).

First of all, the short series of 30 test parts was conducted and analyzed to assess the temperature extraction capability of the lubricants. Figure 12a shows the temperature sensor readings as a percentage of the temperature drop after the production of 30 parts. In accordance with the laboratory tests, the newly formulated lubricant enhanced with an additive to address the Leidenfrost point improves the heat-extraction capacity of the lubricant. It is worth mentioning that for these short production series, the surface die temperature of 450 °C cannot be reached when the lubricant is sprayed due to the limited number of parts produced. Temperature sensors recorded an initial die surface temperature of 90 °C when the lubricant was sprayed. Nevertheless, this short production series confirms the laboratory observed trends and can be used as a rapid screening test to elucidate the effectiveness of different lubricants. Once the higher effectiveness of the newly formulated lubricant enhanced with an additive to address the Leidenfrost point (LUB 2) is proved in an industrial environment, a series of 1200 test parts were conducted only with the reference lubricant and LUB 2. Figure 12b illustrates the temperature sensor readings as a percentage of the temperature drop after the production of 1200 parts. In this case, an initial surface die temperature of 450 °C was achieved. The improvement trend in terms of heat-extraction temperature for LUB 2 is also proven in the long series production.

Surface die wear inspections were conducted during the production of 1200 castings parts. Figure 13 illustrates the evolution of surface die wear at the beginning of the production (polished surface), after the production of 900 aluminum casting parts and at the final stage of the 1200 aluminum casting parts.

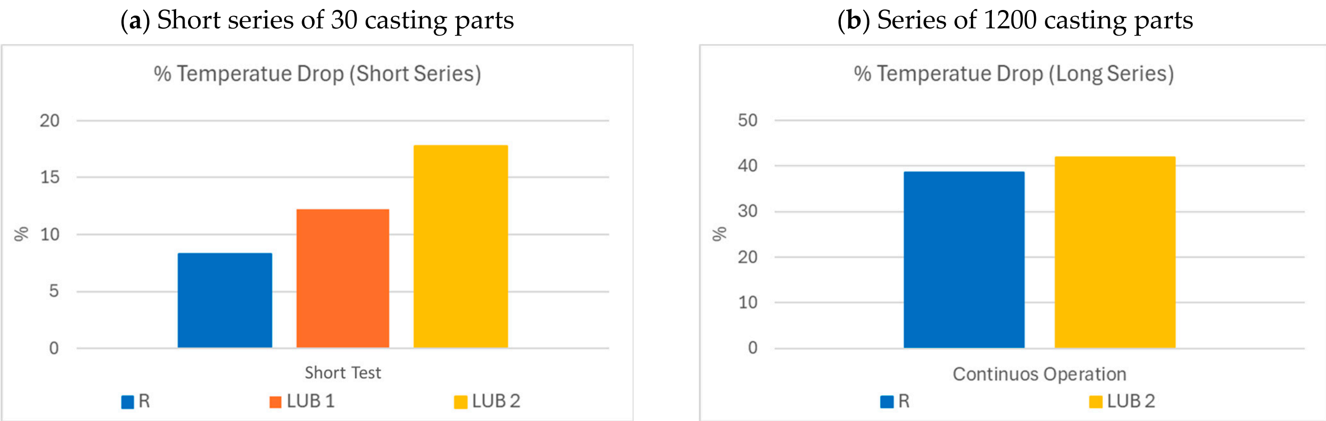

Figure 12. Percentage temperature drop during the (**a**) short series of 30 parts; (**b**) series of 1200 parts for each of the tested lubricants.

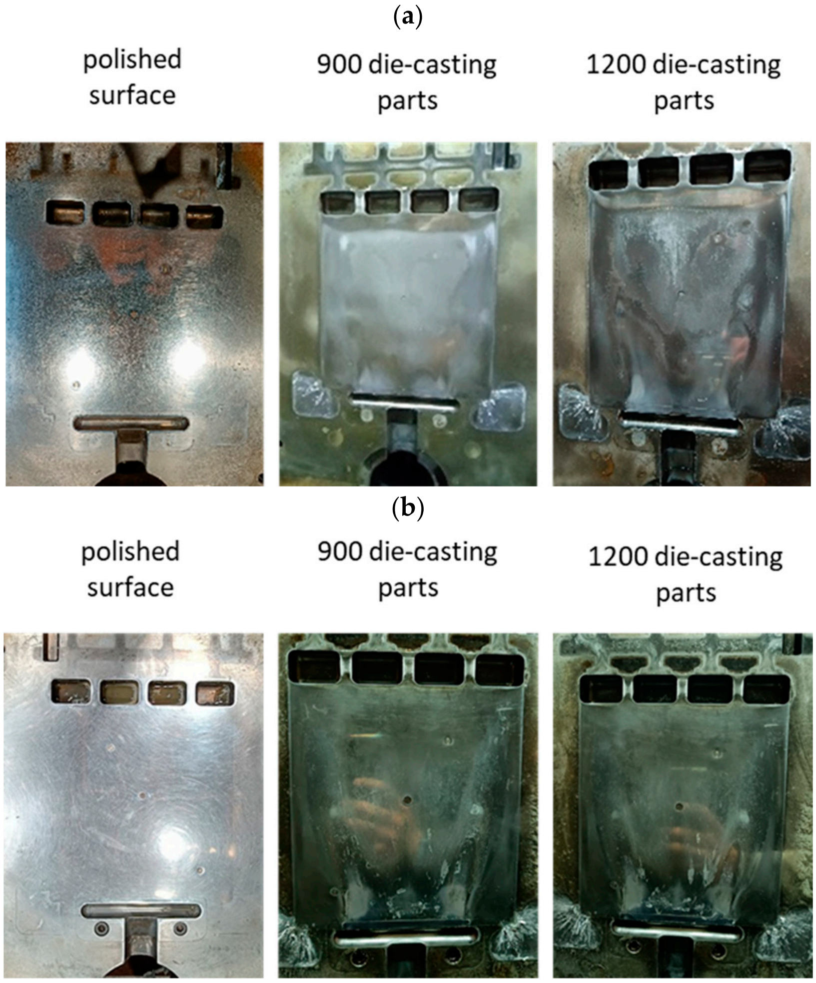

Figure 13. Images of the evolution of surface wear during the production of 1200 aluminum casting parts. (**a**) Reference lubricant; (**b**) newly formulated lubricant enhanced with an additive to address the Leidenfrost point (LUB 2).

Figure 14 shows the 3D topography analysis of the surface replicas applied on the surface die during the wear inspections. It can be observed that the main wear mechanism indentified is adhesion and has a direct effect on the surface roughness. The new lubricant formulated with an anti-Leidenfrost additive appears to be significantly more effective in reducing adhesion and then improving surface die quality.

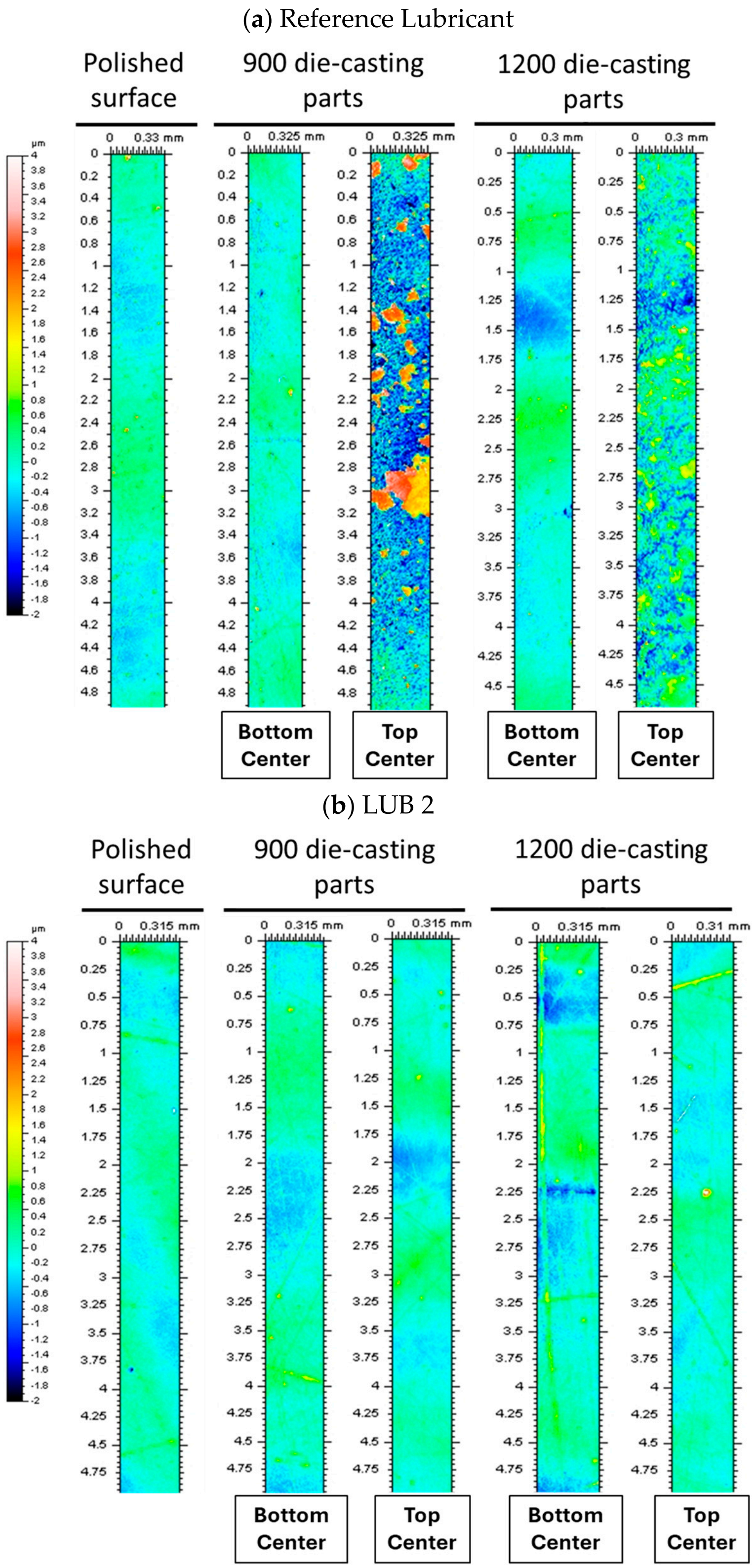

Figure 14. Images of the evolution of surface roughness during the production of 1200 aluminum casting parts.

Figure 15 and Table 6 presents the experimental values obtained for the evaluated surface roughness parameters. Ra and Sa represent the roughness average in 2D and 3D, respectively, while Sdr indicates the texture's contribution relative to a planar area; a perfectly leveled surface has an Sdr of zero, and Sdr becomes larger as the slope of the surface increases. It can be observed that the newly formulated lubricant enhanced with an additive to address the Leidenfrost point (LUB 2) significantly reduces the adhesion in the surface die, thereby reducing the surface roughness of the die, minimizing the wear, and extending the die's service life.

(**a**) (**b**)

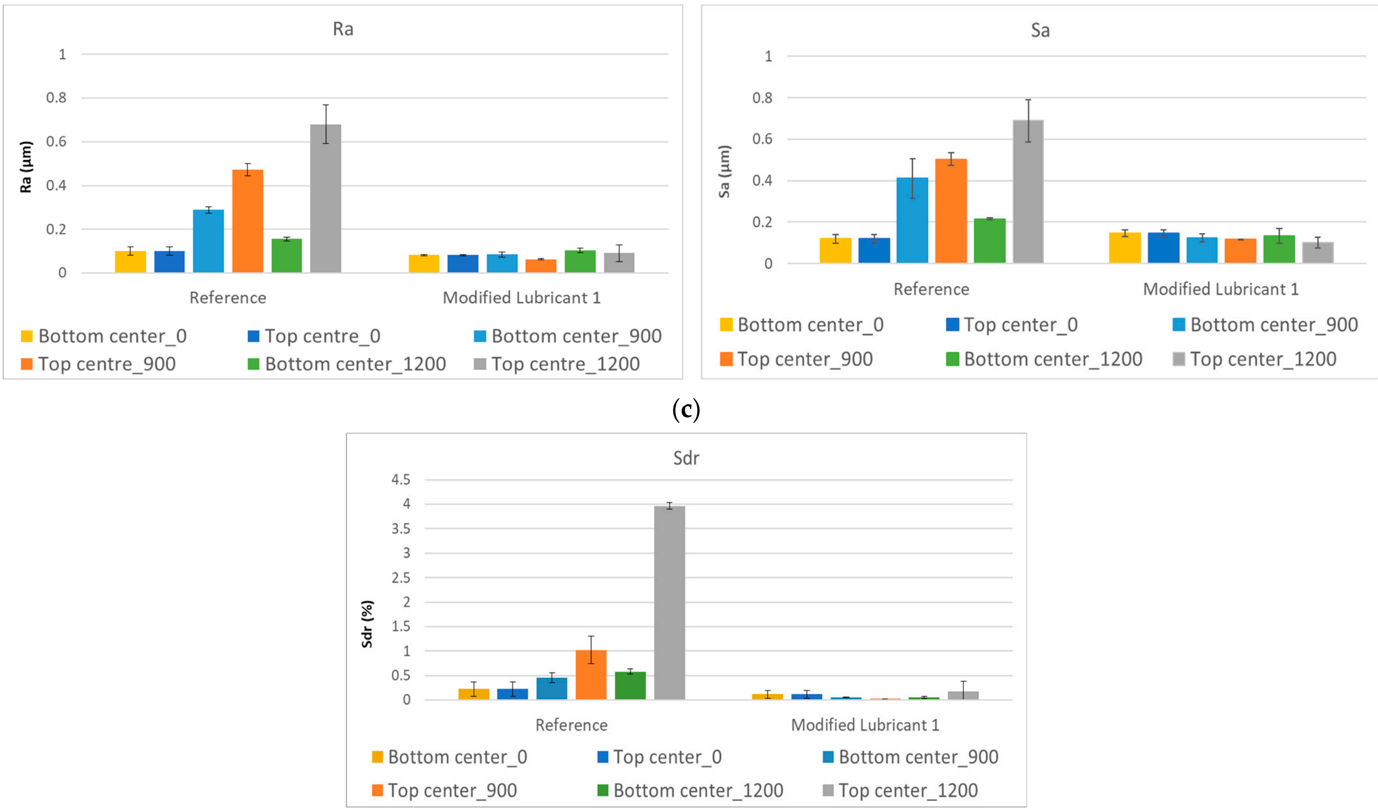

Figure 15. Surface roughness parameters (**a**) Ra, (**b**) Sa and (**c**) Sdr evolution during the production of 1200 aluminum casting parts.

Table 6. Summary of the experimental values obtained for surface roughness parameters.

Lubricant		Zone	Ra [µm]	Sa [µm]	Sdr [%]
Reference	Polished	Bottom center	0.10 ± 0.02	0.12 ± 0.02	0.23 ± 0.15
		Top center	0.10 ± 0.02	0.12 ± 0.02	0.23 ± 0.15
	900 casting parts	Bottom center	0.24 ± 0.11	0.43 ± 0.31	0.45 ± 0.10
		Top center	0.47 ± 0.03	0.50 ± 0.03	1.02 ± 0.28
	1200 casting parts	Bottom center	0.16 ± 0.01	0.22 ± 0.01	0.58± 0.05
		Top center	0.68 ± 0.09	0.69 ± 0.10	3.97 ± 0.06
LUB 2	Polished	Bottom center	0.08 ± 0.01	0.12 ± 0.02	0.23 ± 0.15
		Top center	0.08 ± 0.01	0.12 ± 0.02	0.23 ± 0.15
	900 casting parts	Bottom center	0.08 ± 0.01	0,12 ± 0.02	0.05 ± 0.01
		Top center	0.06 ± 0.01	0.12 ± 0.01	0.02 ± 0.01
	1200 casting parts	Bottom center	0.10 ± 0.01	0.13 ± 0.04	0.05 ± 0.02
		Top center	0.09 ± 0.04	0.10 ± 0.02	0.18 ± 0.21

Finally, the cooling temperature curves recorded during the laboratory test were compared with those obtained from the temperature sensors during the production of 1200 aluminum parts in the pilot plant to validate the heat-transfer capability computed in the laboratory test. Figure 16 illustrates this comparison, showing that the measured temperature curves are consistent. Therefore, the experimental methodology developed for assessing heat-transfer capability is validated as a rapid and cost-effective approach for evaluating lubrication alternatives for HPDC applications.

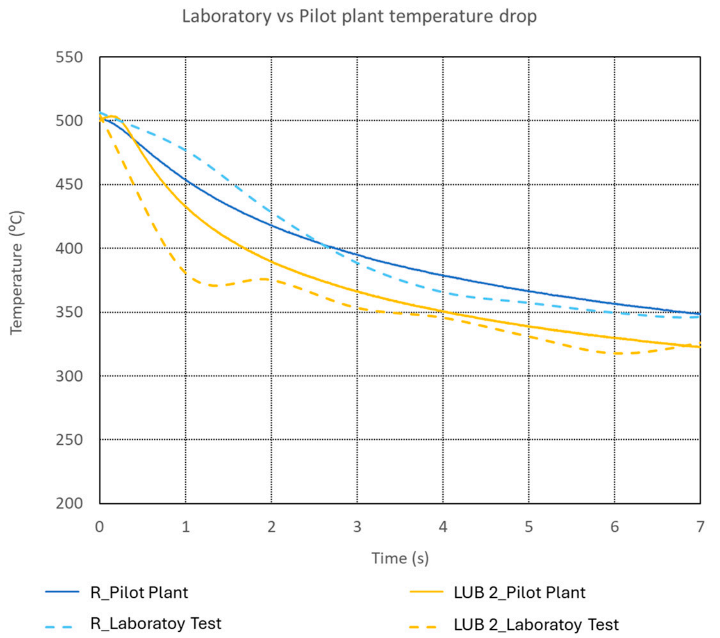

Figure 16. Cooling temperature curves recorded during laboratory (dashed lines) test during the production of 1200 aluminum parts (solid lines) for the reference lubricant and the newly lubricant with enhanced additives to address LFP.

4. Discussion

This work is focused on the study of the efficiency of ester-based lubricants for aluminum high-pressure die-casting applications. Additionally, the effectiveness of a new formulated additive to increase the Leidenfrost point and then decrease aluminum adhesion has been tested. Two different approaches have been considered: laboratory and pilot plant validation. In laboratory test, a modified pin-on-disc test methodology was used to compute the adhesion force of an aluminum ball on a hardened steel sample at 450 °C under different lubricants and in dry conditions. The newly formulated ester-based lubricant (LUB 1) outperformed the commercial mineral oil-based lubricant in terms of aluminum adhesion. It is known that esters improve the lubricity compared to mineral oils [24] due to their interaction with the steel surface, which creates an effective protective layer. The newly formulated ester oil used in the tested lubricants maintained this advantage even at high temperatures like 450 °C, showcasing their potential for use in harsh conditions when they are chemically tailored to meet the industrial needs.

To better understand the influence of additivities to address the Leidenfrost phenomenon in aluminum HDPC, the temperature evolution curve on a hardened steel surface after spraying the lubricant has been measured. In that sense, comparing the results of the commercial lubricant and the new formulated ester-based lubricant, it is observed that the addition of an anti-Leidenfrost additive to the ester-based lubricant significantly improves the results in terms of heat-transfer capacity. Then, the new formulated anti-Leidenfrost additive decreases the Leidenfrost point, which is likely due to the additive increasing surface tension and, consequently, the contact angle. These findings align with previous research that evaluates the relationship between the LFP and CA at high temperatures [23]. However, a direct correlation of the contact angle between the lubricant and the surface and the heat-transfer capacity has not been encountered.

New formulated ester-based lubricants were also tested in an aluminum HPDC pilot plant to validate their performance in an industrial environment. Firstly, a short series of

30 die-casting parts were produced to validate the heat-transfer capacity laboratory results. Although the temperature reached during the test was below the stated industrial die-casting standard and laboratory test parameters, it still was within the range of industrial HPDC die temperatures, with reports indicating a minimum of 80 °C [5]. In that sense, these short series confirmed the laboratory trends validating the methodology to screen the efficiency of new products in terms of heat-transfer capacity. Secondly, a 1200 die-cast series was manufactured to assess the efficiency of the tested lubricants in terms of surface die wear damage. Surface replication techniques were applied to report the aluminum adhesion during the production of the parts. Initially, the surface die was manually polished, and surface inspections were performed at the beginning of the production after 900 produced parts and at the end of the 1200 die-casting parts. The formulation of new anti-Leidenfrost additives in the combination of esters tailored to HPDC applications demonstrates their effectiveness in reducing aluminum adhesion. It has been proved that the newly formulated lubricant enhanced with an additive to address the Leidenfrost point significantly reduces the adhesion in the surface die, thereby reducing the surface roughness of the die and minimizing the wear. In that sense, the tailored formulated lubricant, to meet the aluminum HPDC industry, appears to be an effective and sustainable solution.

Finally, to validate the heat-transfer capability computed in the laboratory test, the cooling temperature curves recorded were compared with those obtained from the temperature sensors during the production of 1200 aluminum parts in the pilot plant. It has been proven that the laboratory measured temperature curves are consistent with the pilot-plant-recorded curves. Consequently, the experimental methodology developed for assessing heat-transfer capability is validated as a rapid and cost-effective approach for evaluating lubrication alternatives for HPDC applications.

5. Conclusions

This work is focused on the validation of newly formulated ester-based lubricants and tailored anti-Leidenfrost additives for the aluminum high-pressure die-casting industry. To validate the lubricants, two different approaches have been conducted. The first approach was based on a newly designed laboratory test. These included measuring the adhesion force (to determine the force needed to break the adhesion betwenn the aliminuim and the 1.2344 hardened steel) and assessing the heat-transfer capability of the lubricants. The second approach was based on a pilot plant methodology that serves as a screening test under industrial conditions and as validation of the laboratory test results. Based on the results obtained in this research, the following conclusions can be drawn:

- A laboratory methodolgy has been established to assess the heat-transfer capability of new formulated lubricants. The methodology has been validated through recorded curves temperatures during pilot plant aluminum die-castings.
- There exists a correlation between heat-transfer capability (linked with the LFP) and aluminum adhesion, while the CA does not directly influence the lubricant performance.
- A pilot plant methodoloy for testing newly formulated lubricants has been established. This methodology validates the efficiency of different formulated lubricants in terms of aluminum adhesion.
- The efficiency of the ester-based lubricant for high-temperature applications, such as HPDC, has been demonstrated. Moreover, the combination of ester-based lubricants with tailored additives to maximize Leidenfrost point has been assessed.

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