Evaluation HPDC Lubricant Spraying for Improved Cooling and Die Protection

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Abstract

This study tries to find out a better cooling and temperature homogenization as well as better die protection on high-pressure die-casting (HPDC) spray lubrication. Test procedures have been set up to study the Leidenfrost point (LFP), contact angle (CA), film thickness and protection from die soldering of lubricants typically applied into the die surfaces during HPDC process.

Five different lubricants have been studied as well as the influence in different controllable process parameters (type of die material, oxidation of the surface, temperature, roughness, droplet diameter, water hardness and lubricant concentration).

The increase of the LFP, avoiding film boiling regime, and a reduced CA, improve the cooling and film ability of die surface during spraying. The best chemistry exhibits high LFP, shows an increased thickness of the formed film and is more effective preventing the sticking of the aluminum part to the die surface. Thermogravimetric analysis shows better thermal properties for lubricants with anti-sticking performance. The study performed and the test protocols set up result in a better insight of the involved phenomena and allow selecting the most favorable operating window for HPDC lubrication.

Keywords:
HPDC, lubrication, Leidenfrost Point, Contact Angle, Film Thickness, Die soldering, thermogravimetry

1 INTRODUCTION

High-pressure die-casting is a single step process producing accurate, thin walled and very detailed light alloy parts at high productivity. Typically molten Al alloy is injected into a steel die at high speed and solidified under high pressure. When the solidification is completed and the casting cooled sufficiently, the die is opened and the diecasting is mechanically ejected by activated pins. The die surfaces are then cooled by lubricant spraying [1]. Die lubricants are sprayed onto the inner contour of the die cavities for many reasons [2]:

- to cool and balance the die temperature;
- to create a lubricating film that facilitates the filling and the extraction of the part acting as release agent;
- to create a protecting film that, together with the surface oxide layer, prevents die soldering phenomena.

The temperature variation on the die surface during spraying operation ranges from a maximum of 300ºC to a minimum of 80ºC [3]. In this range, the heat flux depends on the boiling regime as shown in Figure 1. In order to achieve optimum cooling conditions, the die surface should be able to work at temperatures below the LFP, where the heat transfer is higher. At the LFP, a vapour film forms a continuous barrier over the die surface that lubricant cannot penetrate, and the formation of the protective film that prevents die soldering is not formed. The *wettability* is the ability of a liquid to cover a solid surface, assuring full contact. This physical property is measured by the CA between the die surface and the lubricant droplet. Covering a larger area through reduced contact angle, the heat transfer capacity is improved and a wider lubricant film is formed at the die surface.

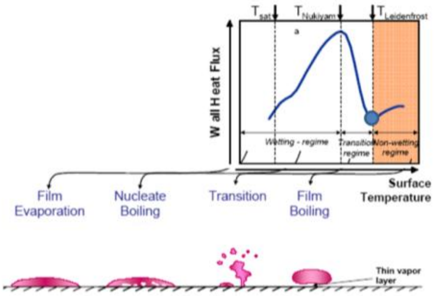


Figure 1: The boiling curve of a droplet, where the heat flux dependence on the surface temperature due to the boiling regime is shown [4].

Once the correct filming of the die surface is achieved, the chemistry and the thickness of the film are also important on the performance as release agent and protective film against die soldering. Other requirements can be requested to lubricants, for example they don't have to disturb post processes of the castings such as welding or sticking with glue, but these aspects have not been studied.

In this study, a new testing device has been setup to measure the LFP and the CA in order to understand individually the effect of different surface status (materials, roughness, oxidation), lubricant characteristics (mineral, synthetic, dilution, water hardness), temperature, spray properties (nozzle diameter). In the protocol setup, the measured LFP differs slightly from the one in HPDC, but this procedure provides certainly

independent, relative and valuable information on the nature of this phenomenon. For evaluation of the effectiveness of the formed film, film thickness measurements have been carried out, and a tribological test to evaluate the capability of the lubricant to prevent die soldering and the frictional behaviour as release agent has been set up. Finally, differential scanning calorimetry and thermogravimetric (DSC/TGA) tests have been performed to the lubricants.
The results and test procedures are expected to be useful for improving the lubricant selection and film formation, the cooling and the temperature homogenization of the spraying process during HPDC.

2 MATERIALS AND METHODS

2.1 Materials

Five different lubricants for HPDC applications have been used for this study, supplied by Motul-Baraldi in the form of concentrated lubricant and hereafter named A, B, C, D, E. The lubricants are generated from a concentrated solution composed by a 20-30% of an anhydrous part that is usually diluted in water once time more with a rate from 1:50 to 1:100 [2]. The active part, made of a blending of oils, polysiloxanes, polymers and many additives, gives the main characteristics to the film that forms on the die cavity after water vaporization. Together with the anhydrous part, there are the emulsifiers, normally surfactants, which have the aim to join the oil and water and to increase the filming power on the die surface [5].
Based on the typical classification of metal working fluids [6], the B, D and E lubricants could be classified as semi-synthetic lubricants, since they contain mineral oil with a viscosity index estimated around 90; the A and C lubricants are synthetic ones with a viscosity index estimated around 180. The A and C lubricants offer a very high releasing power for average to big weight diecastings. The B lubricant is particularly indicated in the case of thin walled components with a low weight, and the D and E lubricants for general purpose, not recommended to any specific kind of diecast parts.
The reference dilution was 1:50 (2%) but other ratios were studied as well, from 1% up to 20%. The surface tension of lubricants diluted at 1:50 was measured according to the ASTM D-971-99 standard. The dilution were made mostly with tap water (7.52 ºdH German grade, 53.6 mg/l Ca^{+2}, <1 mg/l Mg^{2+}) and artificially generated hard water (17.9 ºdH, 96.8 mg/l Ca^{+2}, 18.7 mg/l Mg^{2+}).
Table 1 shows the measured surface tension values for the analysed lubricants. It is important to highlight that these HPDC lubricants at such a small dilution rate as 2% drastically reduce the water surface tension from 72 mN/m to around the half of this value.

Lubricant	Surface Tension (mN/m)
A	48.7
B	53.2
C	35.1
D	33.9
E	31.5

Table 1. Measured surface tension of the analysed lubricants at 2% dilution ratio.

Two different type of steels, AISI H13 and H11, were selected for the study, since they are the most common die materials in HPDC [7].

2.2 Leidenfrost point and contact angle measurement

Experimental measurements were carried out by a SURFTENS universal goniometer shown in Figure 2, which was used for the determination of the LFP, as well as the CA. The apparatus used here is similar to that described in [8] used for the similar purpose, but its main advantage is the capability of measuring both the LFP and CA. The universal goniometer was modified by IK4-Tekniker from original version to be able to measure CA at high temperature and the LFP.

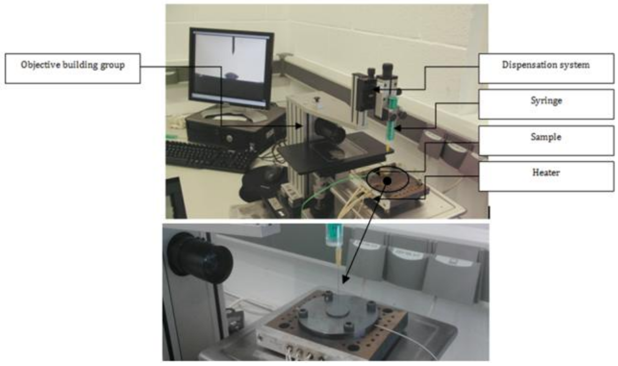


Figure 2. The universal goniometer modified by IK4-TEKNIKER to carry out high temperature measurements.

The equipment is composed by a fixed structure with an objective building group, an illumination unit and a droplet dispensation system, which was here used with two different nozzle diameters, 0.5 and 0.9 mm respectively. Most of cases were performed with the 0.5 mm diameter, except for dedicated tests used to study the influence of the droplet size. The sample is located over an electrical resistance heater fixed to a mobile structure. In order to check the temperature, a K-type thermocouple is placed into the sample and connected to a control unit.
The criterion used to identify the LFP was to increase the temperature progressively, in order to identify the temperature when the film boiling regime began. A screening of the lubricant impact behaviour at the surface was recorded. After each droplet falls to the hot surface, the sample is rotated to carry out further test in a new area that is not contaminated by previous lubricant droplets.
The same system is used to evaluate the contact angle. When the droplet falls from the dispensation system onto the surface, automatic measurement and data recording of CA is made from identification of 5 points of the droplet shape, and a CA value is reported as the mean given by the data acquired during 30 seconds. Each measurement was repeated at least four times. The heating device was used to heat the surface at 25, 50 and 80ºC, respectively, in order to evaluate the CA at different temperatures.

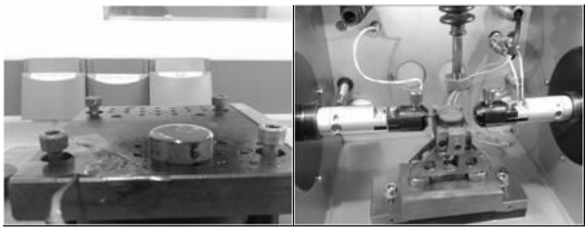
2.3 Die soldering protection measurement

A reciprocating pin-on-disc test, where a cylindrical pin of aluminium EN-AC-43500 is forced to move against a mould material in AISI H13 steel disc, has been used for evaluating the performance of lubricants to prevent soldering and as release agent.

To create good conditions to simulate the presence of the lubricant film on the disc surface, a procedure to create a lubricant coating on the disc surface has been set up, based on resistance heating. About 1 ml of pure lubricant was progressively deposited on the H13 surface at 130ºC, resulting in a homogeneous and well distributed lubricant film.

The machine used is a SRV©. The testing temperature is selected as close as possible at HPDC die surface. A low temperature condition (~365ºC) has been selected to study the phenomena in a range of temperature that is equal to the average surface temperature in real HPDC process, and a higher temperature (~450ºC), which is the maximum value of the die temperature during die filling.

The basic normal load and sliding speed selected to reproduce the wear mechanism were 50 N and 25 Hz. The stroke was kept constant at 2 mm. To investigate this effect, different level of pressure and speed were considered: 20-50-125-200-300 N and frequencies of 10-25-35Hz.



(a) (b)

Figure 3. (a) Preparation process of the sample with a ''coating'' of the lubricant and (b) sample that is ready to start the pin on disk test

In order to evaluate the effectiveness as release agent, the stable initial value of friction was considered. It was not not identified a big difference between lubricants friction behavior as shown in the Table 2. As output for die soldering prevention the time to lubricant failure has been identified to be good indicator of lubricant effectiveness. The failure is measured when the friction coefficient rises from the stable value of 0.1-0.2 to a value of around 1 of friction coefficient.

2.4 Film thickness measurement

The procedure for film thickness formation of lubricants is quite simple. A lubricant film is formed similarly to the samples of pin-on-disc tests and leaving drying for 5 hours. Afterwards a general tool for measuring thickness's of non-magnetic coatings, EASY-CHECK FN from NEURTEK Instruments, is used for thickness measurement, shown in Figure 4. About 20 measurements are taken in different points of the sample surface.

(a) (b)

Figure 4. (a) Steel sample coated with a lubricant film (a) and (b) thickness measurement device

2.5 DSC/TGA measurement

This test has been made with a pure lubricant with an initial mass equal to 40 mg. Different lubricants have been tested to evaluate the effect of the temperature in lubricant degradation.

The range of temperatures investigated in the dynamic thermal test were from 50 up to 700°C. The heating rate was 10°C/min using N_2 atmosphere. Isothermal tests have been performed in the range from 350 to 425ºC.

Figure 5. Lubricants ready to be tested in the DSC/TGA test

3 RESULTS AND DISCUSSION

3.1 LFP and CA measurements

LFP and CA for different water based lubricants in H11 and H13 hot work tool steels

Tests to search LFP were carried out with liquids diluted in soft water, both for H13 and H11 samples. The results are reported in Figure 5.

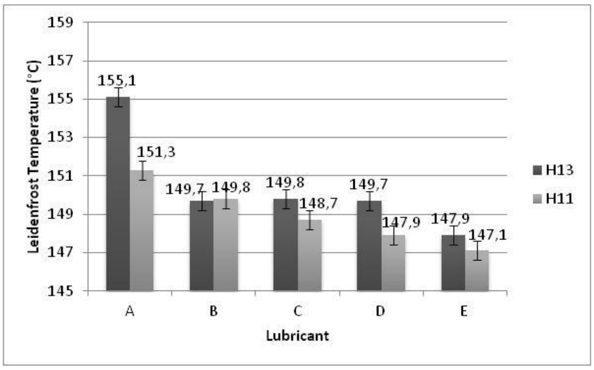


Figure 5. The LFP for soft water-based lubricants on H13 and H11 die materials.

The differences between lubricants, on the value of LFP, are probably due to the different formulations and additives. For both die materials, lubricant A has the highest LFP and lubricant E has the lowest LFP, while B, C and D are similar. The LFP is typically higher for H13 for most of the lubricants.

Regarding the CA values for different lubricants, shown in Figure 6, there is a wide range of values from almost 70º to below 15º depending on lubricant and surface temperature. It is shown, a reduction of contact angle when increasing the temperature, but not all lubricants reduce their values in the same way. The synthetic lubricants A and C, have less differences in contact angle at different temperatures with both steels, H13 and H11. This is probably due to their higher viscosity index due to their synthetic nature that means a lower dependence of the viscosity with the temperature. All the semisynthetic lubricants have a similar behavior.

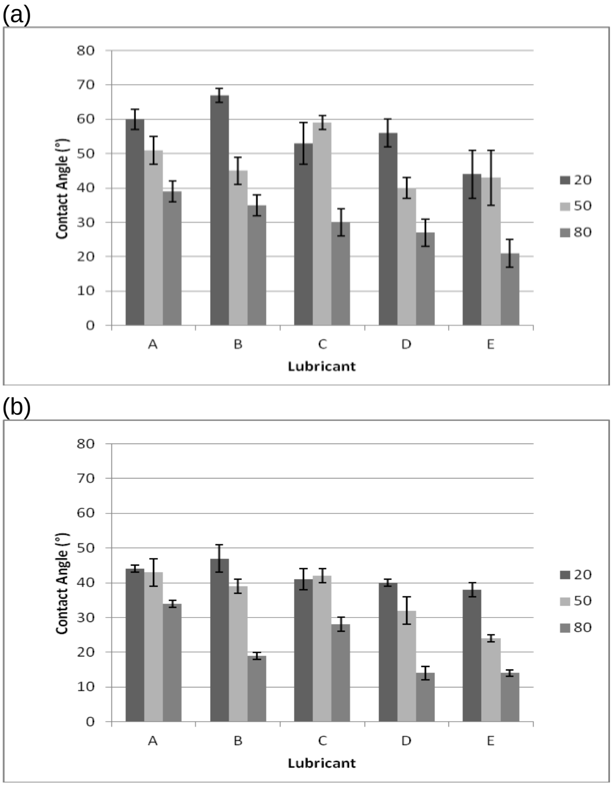


Figure 6. The CA at 20, 50 and 80°C on (a) H13 and (b) H11 steels

The trend of these curves highlight that the LFP decreases with the decrease of contact angle (CA). An explanation for this phenomena could be that a less wettable liquid has less contact area with the surface, but its weight is the same. Thus, the higher pressure makes necessary a thicker vapour layer to sustain the droplet, then this layer will be created at higher temperature.

Study of effect of different parameters on LFP and CA

A study of the different variables that affect LFP and CA was done and the results can be here summarized.

- The variation range of the LFP for all the studied cases is in the range 140-165ºC. These differences are significant for HPDC.
- The variation range of contact angle for all the studied cases is in the range 15-70 degrees; the repetitions show up to 5 degrees of deviation.
- The lubricant ranking of LFP for all the cases shows: highest value for A, lowest value for E and in the middle similar values for B, C and D.
- The higher the temperature, the lower is the contact angle. In general the lubricant CA values at 80ºC present a similar behaviour to temperatures near LFP.
- The CA decrease is less pronounced for some lubricants. The synthetic lubricants A and C, have a lower variation of CA with temperature, probably due to the higher viscosity index of this type of lubricants.
- The lubricant ranking of CA, even if it is not the same ranking for the 3 tested temperatures, is similar to the LFP ranking. In fact, more hydrophobic combinations (higher contact angle) tend to have higher LFP.
- The LFP and CA for H13 are a bit higher than H11 probably due to their finer grain microstructure.
- The hardness of the water used for the formulation of the lubricant has some influence in the LFP for both steels H13 and H11. In general, the harder the water, the higher was the LFP being this behaviour especially significant for lubricants C and D.
- Oxidized surfaces show higher LFP, since the oxide layer, has a lower thermal conductivity that promotes surface temperature protection.
- A higher lubricant percentage (1-3%) leads to higher LFP. Above 3%, it is not observed further increase.
- Rougher surfaces have higher LFP both for H13 and H11 steels, but the CA variation depends on the type of steel and their microstructure. In the H13 with a finer microstructure, the contact angle increases with the roughness, they present a hydrophobic behaviour and it seems to fulfil Cassie's model [12] where the liquid doesn't penetrate the roughness. The H11 seems to follow the Wenzel's model [12] range where liquid penetrates in roughness grooves, and the contact angle decrease when increasing the roughness.
- Higher droplet size shows higher LFP, in line with prediction from We number [11]. Wettability seems not to be influenced by droplet size, being the droplet size, a parameter that could play a good role trying to maximize LFP keeping low the contact angle.
- Die cast lubricants get lower surface tension, CA and LFP values than water. The droplet of the lubricant flattens after impact and then it evaporates, while water evaporates without an increase of surface contact.
- The combination of several factors make synergic the improvement, increasing further LFP. An increase of 25ºC in LFP relative to the reference, were achieved combining a high droplet size 0,9mm diameter, with 3% of lubricant A, diluted on hard water, on an oxidized sample, with high roughness.

3.2 Time to film failure on pin-on-disc tests

The lubricant behaviour and its tendency to be washed from the steel surface depend on many factors such as: pressure, speed, temperature and surface conditions. The main parameter that affects this tendency isn't a physical variable but it's a chemical parameter. The chemical composition (especially the amount and typology of additives) describes the desorption tendency of the lubricant.

Regarding the physical parameters, as it is shown in the response surface created from experimental results shown in Figure 7, temperature was the most important parameter (as exponential dependence), followed by Pressure (logarithmic dependence), while the speed didn't show any influence. This is in accordance with Fick diffusion law, which also rules die soldering mechanism [21].

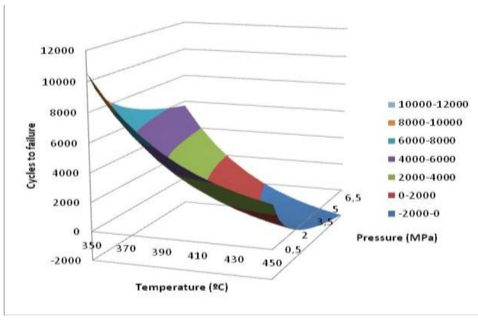


Figure 7. Surface response of time to failure for the A lubricant dependent of Temperature and Pressure.

The time to failure ranking of lubricants was similar to the one of LFP. The table 2 shows these results, being A lubricant the one that lasted more followed by C and D, while B and E where the worst.

	A	C	D	E	B
Time to Failure (cycles)	39750	21625	16025	6500	5000
COF (-)	0,167	0,166	0,177	0,155	0,187

Table 2. Time to failure and friction coefficient of the different lubricants in the pin on disc tests at 50N, 25Hz and 365ºC

3.3 Film thickness measurements

The resulting film thicknesses are shown in Figure 8. The scattering of the data is quite big because the film thickness is not totally flat, since it is a curvature of the film and have some irregularities. The mean value shows an important difference between the lubricants. The lubricant A had the highest film thickness (400µm), followed by C (~300µm), D (~250µm), while B and E, have the thinner thickness (<200 µm).

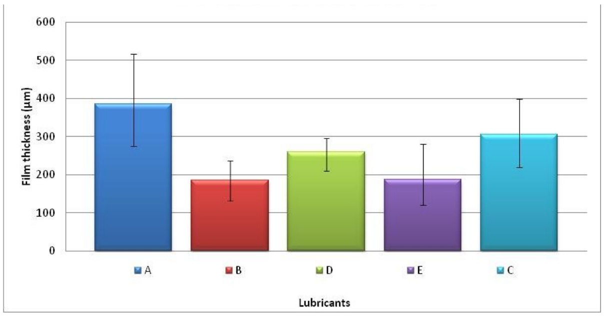


Figure 8. Results of film thickness measurement

3.4 TGA tests

It has been observed that the main element that affects the degradation mechanism of the protective film is the temperature; it leads to an increasing desorption tendency of the lubricant and a chemical degradation of it. These two aspects have been studied with another chemical test, the TGA test. This TGA test has been divided in two different parts: the isothermal and thermal dynamic tests. The first one has the scope to evaluate the effect of the time at high temperature conditions, and the second one has the aim of evaluating the effect during temperature variation.

The dynamic tests have shown that the lubricant has a very high initial degradation (at 100°C) because of the evaporation process. The degradation doesn't carry on until 375°C; above this temperature there's a high degradation process that causes a complete degradation of lubricant above 475°C. In this degradation process it is a difference between the different lubricants (Figure 9).

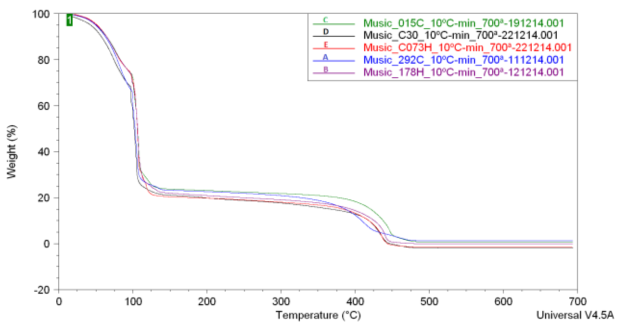


Figure 9. Results of non isothermal / dynamic test of the analysed lubricants

The isothermal test of the lubricant have been carried out at 350°C and at this temperature, after 3,5 hours, the degradation process isn't complete. At 425°C (Figure 10) the total degradation of the lubricant appears after about 30 minutes of test. The ranking of degradation process in isothermal tests and dynamic tests was the same, and as it is as well coincident, with the already stated at film thickness measurements.

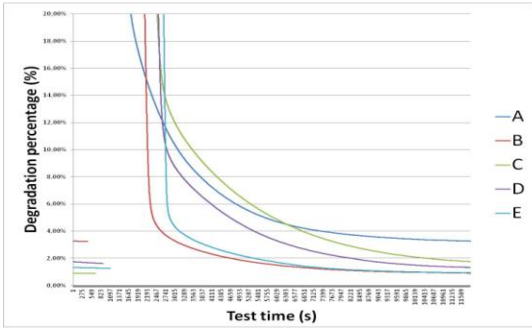


Figure 10. Results of isothermal DSC-TGA tests at 425ºC carried out on the analysed lubricants

4 CONCLUSIONS

Taking into account the requirements of lubricants used for the spraying of HPDC die surfaces, different experimental procedures have been set up. It included Leidenfrost point, contact angle and film thickness measurements for the evaluation of filming capacity and effectiveness on heat extraction. Effectiveness for die soldering prevention was tested through pin-on-disc tests. Termogravimetric TGA tests were performed to evaluate the thermal behaviour of lubricants.

Good correlation was found between filming capacity (Leidenfrost point, contact angle stability at high temperature, film thickness), TGA of lubricants (thermal behaviour) and time to failure during pin-on-disc tests (die soldering prevention).

In these tests the best lubricant is the A followed by the C, both lubricants contains in their formulation synthetic oils with a high viscosity index. Lubricants D, B, and E in this order follow in the best performance ranking.

Isothermal TGA test at 425ºC has been found as a suitable test for screening of big amount of lubricant formulations, which will give an idea of their behaviour on the mould, due to the correlation found between the different tests. The rest of the tests can be used for more detailed studies or when the evaluation of a specific characteristic of the lubricant (i.e. high LFP or high die soldering protection) is needed.

5 ACKNOWLEDGMENTS

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6 REFERENCES

[1] F. Bonollo, N. Gramegna, The MUSIC guide to key-parameters in High Pressure Die Casting, ISBN 978-88-87786-10-1.

[2] L. Andreoni, M. Casè, G. Pomesano, "Lubrificazione della cavità dello stampo", Quaderni della colata a pressione delle leghe di alluminio 7, 1996, pp.9-26.

[3] J.L. Graff, L.H. Kallien, "The effect of die lubricant spray on the thermal balance of dies", Paper T93-083, NADCA Meeting, Cleveland, 1993

[4] Simultaneous Measurements of droplet characteristics and surface thermal behaviour to study spray cooling with pulsed sprays" by Humberto M Loureiro, Miguel R A Panao, Antonio Luis L Moreira of Mechanical Engg Dept Instituto Superior Tecnico at Lisbon Portugal.

[5] L. Baraldi, C. Raone, "Relazione tra la lubrificazione e lo stampo - Lubrificazione in funzione delle fasi di processo", La Metallurgia Italiana 3, 2004, p.37

[6] P.Bittorf, S.G.Kapoor, R.E.DeVor, "Transiently Stable Emulsions for Metalworking Fluids", Department of Mechanical Science and Engineering University of Illinois at Urbana-Champaign, December 2011

[7] L. Andreoni, M. Casè, G. Pomesano, "Il lavoro termico dello stampo", Quaderni della colata a pressione delle leghe di alluminio 5, 1995, p.9.

[8] N. Nagai, S. Nishio, "Leidenfrost temperature on an extremely smooth surface", Exp. Therm. Fluid Sci.12, 1996, pp.373-379

[9] K. Domkin, J.H. Hattel, J. Thorborg, Modeling of high temperature- and diffusion-controlled die soldering in aluminum high pressure die casting, Journal of Materials Processing Technology 209 (2009) 4051–4061

[12] J. C. Berg, "Wettability", Ed. Marcel Dekker, ISBN: 0824790464.

[11] J.D. Bernardin, C.J. Stebbins, I. Mudawar "Mapping of impact and heat transfer regimes of water drops impinging on a polished surface", Int. J. Heat Mass Transfer 40, 1997, pp.247-267