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Development of a Laboratory-Scale Test Methodology for Performance Evaluation of Lubricants for Hot Stamping of an Aluminium Alloy

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Abstract: In hot stamping of aluminium, the need for efficient methods to evaluate, compare, and rank lubricants based on their tribological performance is critical in the early stages of selection. Pilot and simulative testing can be costly, time-consuming, and complex, making it inefficient for initial benchmarking. This work aims to develop a test methodology to assess lubricant performance for hot stamping under key operating conditions without fully simulating the forming process. The proposed method distinguishes the impact of temperature on lubricant degradation, friction, wear response, and cleanability. The tests utilised a conventional hot work tool steel and a 6010S aluminium alloy with two commercially available lubricants: a polymeric lubricant and a lubricant containing graphite. The tribological tests involved a reciprocating, sliding flat-on-flat configuration at two temperatures (100 °C and 300 °C). The methodology showed that the graphite-containing lubricant exhibited over a four times lower friction coefficient than the polymer-based lubricant at 10 wt.% concentration and 300 °C. At 100 °C, both lubricants provide lubrication and can be cleaned, but increasing temperature led to a significant decline of both aspects. The observed temperature range where the lubricants degrade was between 120 °C and 170 °C.

Keywords: aluminium hot stamping; lubricants for hot stamping; lubricant cleanability

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1. Introduction

The use of aluminium alloys has increased in recent years in the automotive industry due to their excellent strength-to-weight ratio, energy absorption capacity, and high recyclability. Due to their formability limitations at room temperature and their spring-back effect, high-strength aluminium alloys are often formed at a high temperature [1–3]. Hot stamping of aluminium is on the rise because it can achieve complex shapes and control the mechanical properties of the final component.

The operating process conditions are known to cause abrasion, adhesion, and, more critically, material transfer from the aluminium onto the tool surface [2,4–7]. Lubricants are used in aluminium forming to alleviate these problems. Solid lubricants, such as graphite, molybdenum disulphide, and boron compounds, are commonly used for hot stamping of aluminium due to their ability to reduce shear stresses in sliding contacts [4,6,8]. Other solid lubricants that have recently surfaced are in the form of additives in oil or water emulsions [9]. Polymer-based lubricants have also gained interest thanks to their potential to finetune the lubricating properties by combining different compounds [4,8,10]. As new lubricant solutions emerge, uncertainties regarding their performance in hot forming also arise.

The cleanability of the lubricants from the workpiece and the forming tool is an aspect that is not commonly reported, or that is only briefly mentioned on online catalogs, which is in part due to the continuous development of lubricants and in part because focus is given to their tribological behaviour. Cleanability is an important factor for quality and process efficiency, as well as downstream processes such as heat treatments, painting, or welding. Furthermore, the accumulation of lubricant on the die over time affects the process efficiency and can result in necking if the coefficient of friction reduces too much due to excessive lubrication [11,12]. Krajewski and Morales observed that when tools were lubricated, the propensity for necking was increased compared to unlubricated tools. However, expectedly, the tool was prone to galling when no lubricant was used. There is clearly a balance between the amount of lubricant that should be on the tool to control friction and wear; thus, it becomes critical to ensure that lubricant can be maintained at an optimal level. Many studies highlight the negative health effects of synthetic lubricants, or of dry lubricants using small particles. In a review by Opia et al. [13], they review the many benefits and potential of using bio-based lubricants in different applications, highlighting their biodegradability and low hazard properties. However, as almost every study, no information is given regarding cleanability aspects of lubricants. Thus, it is critical to address this knowledge gap in a systematic manner.

A critical challenge in the development and validation of lubricants for hot forming is the relatively limited options available for assessing and ranking the lubricants' performance in conditions similar to the forming operation. The number of tribometers designed to simulate sliding conditions in hot stamping is limited, and some of the most salient drawbacks with these is their complexity (which is necessary to closely simulate the contact conditions), their need for significant costs, and time for preparation and operation, making screening tests or exploratory studies difficult and not economically viable. Simple benchmark test equipment exists and is used in many studies, but these studies usually focus on cold forming, evaluate lubrication at low temperatures, or focus on different materials than aluminium. Some studies do exist where simple benchmark tests have been developed to evaluate lubricants. Noder et al. [14] conducted tests using twist compression tests to assess the performance of the lubricant in warm forming applications. They were able to rank different lubricants as a function of temperature and sliding distance before lubricant film failure. This type of test enables the relatively easy ranking of the lubricants, but has the clear disadvantage of not being representative of actual friction values or distance before lubricant failure. In a study by Shafiee Sabet et al. [15], a relatively simple contact configuration was used to replicate the contact conditions experienced in the forming of aluminium and evaluate lubricant behaviour at temperatures up to 100 °C. This tribometer has the advantage of using small samples and not being resource demanding, but it is not as widely spread and common in most testing facilities and it is unclear what the maximum temperature attainable is. There is a need to develop test methodologies using easily or readily available test equipment with simple configurations, which offers significant value when developing new lubricants for hot stamping of aluminium, because demand and interest is significant.

In this study, one of the primary objectives is to develop a simple test methodology using a reciprocating friction and wear tester to evaluate lubricants used in hot stamping of aluminium. The applicability of this method focuses not on simulating the forming operation, but on selecting parameters relevant to the hot stamping of aluminium that affect the lubricant performance, such as temperature. Using the aforementioned method, this work aims to compare the friction and wear behaviour of two different lubricants developed for aluminium hot stamping, and evaluates the effect of lubricant concentration and temperature on their tribological response. Furthermore, this study also explores cleanability aspects of these lubricants as a function of temperature. This is a critical aspect of lubricants that is significantly under-reported in the open literature and that is critical when evaluating lubricants for hot sheet metal forming.

2. Experimental Procedure

2.1. Materials and Specimens

A commercially available pre-hardened hot work tool steel, Toolox 33, was used for the tribological tests, and the counter surface was an aluminium 6010S alloy provided by Norsk Hydro (Finspång, Sweden). The chemical composition of the materials is shown in Table 1, as provided by the suppliers. The tool steel pin specimens were ground with a defined surface lay, to an arithmetic surface roughness (Sa) of 0.15 ± 0.03 µm. The aluminium alloy was used in the as-delivered condition, which also has a defined surface lay and a Sa of 0.46 ± 0.02 µm after the manufacturing process. The surface roughness of the specimens was measured in the lab by means of white light interferometry.

Table 1. Chemical composition of the tested materials in wt.%.

Aluminium Alloy Composition								
Cr	**Cu**	**Fe**	**Mg**	**Mn**	**Si**	**Ti**	**Zn**	
0.01	0.23	0.30	0.76	0.53	0.94	0.024	0.19	-
Tool Steel Composition								
C	**Si**	**Mn**	**P**	**S**	**Cr**	**Mo**	**V**	**Ni**
0.22–0.24	0.6–1.1	0.8	Max. 0.010	Max. 0.002	1.0–1.2	0.3	0.10–0.11	Max. 1.0

The lubricants used in this study were commercially available lubricants; one was a polymer-based water emulsion with a free release agent and white appearance. The second lubricant was a graphite-based forging emulsion with a black appearance. Both lubricants are designed for hot and warm forging of ferrous and non-ferrous metals.

2.2. Tribological Equipment and Test Methodology

The tribological test set-up was a reciprocating sliding friction and wear tester (Optimol® SRV III); a schematic representation of the contact configuration is shown in Figure 1a. The tribometer consists of an upper specimen (tool steel pin) that oscillates by means of an electromagnetic drive against a stationary lower specimen (aluminium alloy plate). The test configuration was a pin-on-plate configuration, resulting in flat-on-flat contact. The tool steel pin specimens were cylinders with an end diameter of Ø 4 mm and the aluminium alloy was in the form of a plate of 20 mm × 20 mm × 3.5 mm; representations of the specimens are shown in Figure 1b. The tribological tests were performed with a perpendicular surface lay between the pin and the plate, where the orientation of the tool steel topography was parallel to the sliding direction (Figure 1b).

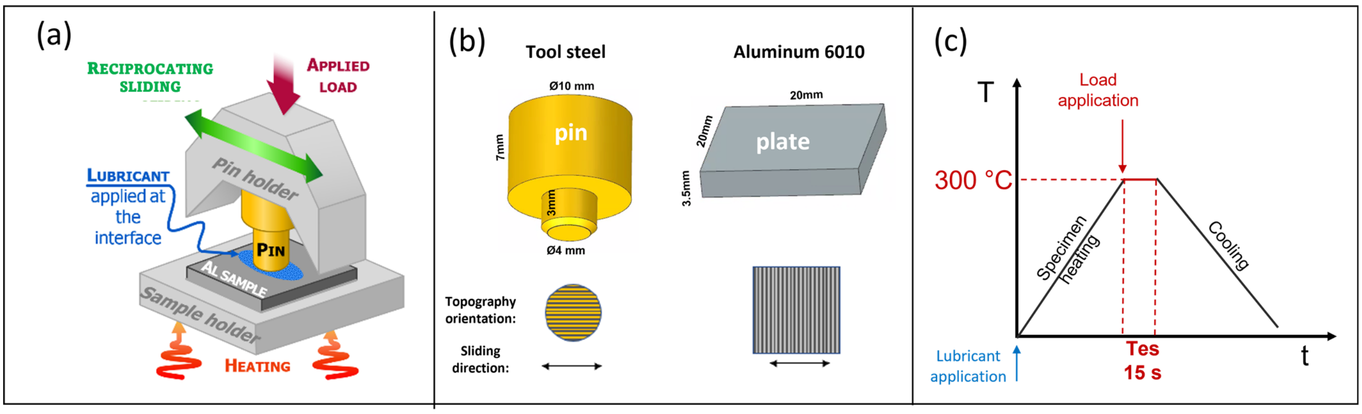

Figure 1. (**a**) Schematic representation of the test set-up, (**b**) surface sliding configuration, and (**c**) heat cycle.

The aluminium alloy plate is mounted on a heating block that is actively heated up. The friction coefficient is measured throughout the entire duration of the test and the tribometer controls, and monitors the sliding frequency, stroke length, load, and temperature.

For the tests, the lubricant was applied on both specimens and left to dry at room temperature. The lubricant was applied with a pipette to measure the quantity and to apply the same volume in every test. One drop was applied on the tool steel pin and 1 mL on the aluminium alloy plate. The lubricants were diluted in distilled water and the concentrations used were 10 wt.%, 50 wt.%; and 100 wt.% concentration.

After applying and drying of the lubricant, the heating cycle was initiated. During the initial heating, the specimens were kept separated and the load was applied only after the desired temperature was reached. Table 2 shows the test parameters. A schematic representation of the heat cycle is shown in Figure 1c.

Table 2. Tribological test parameters.

Parameter	Value
Temperature	100 °C and 300 °C
Load	10 N
Contact pressure	0.8 MPa
Stroke length	4 mm
Frequency	12.5 Hz
Test duration	15 s
Sliding distance	1500 mm

It is important to note that the contact pressure used in the current investigation was selected to promote as minimal ploughing as possible on the hot aluminium alloy. In the forming operation, contact pressures are typically higher, particularly at the radii of the forming dies. The contact pressure in the current study can consequently be viewed as a mild condition, which can be experienced in the flat regions within a forming die.

2.3. Analysis Techniques

The wear mechanisms, surface damage, and cleanability were analysed using a Dino-lite Optical Microscope (AnMo Electronics Corporation, Taiwan), as well as a Zeiss Merlin FEG Scanning Electron Microscope (SEM), Germany, coupled with energy dispersive spectroscopy (EDS).

The surfaces of the specimens prior to the tribological tests were analysed by means of a 3D white-light optical interferometer Ametek Zygo NewView 9000, USA, using a 10× objective lens with a 0.83 mm × 0.83 mm analysis area. Samples were analysed at random, and 5 values were averaged for each material.

Additionally, the lubricants were characterised by means of differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) using a simultaneous thermal analyser instrument. The undiluted lubricants were analysed in the air atmosphere using Pt crucibles from room temperature up to 300 °C and a heating rate of 10 K/min.

2.4. Cleanability Analysis Methodology

A qualitative study was conducted to evaluate the cleanability of the lubricants. The cleaning evaluation made use of different methods after exposing the specimens at 150 °C and 300 °C for 6 min using the undiluted lubricants. These temperatures were chosen based on the DSC/TGA analysis and the observed changes.

The methodologies were developed using a mixture of different cleaning methodologies such as mechanical, dissolution, and chemical cleaning, as well as using different agents such as detergent and ethanol. Figure 2 shows the three different cleaning methodologies.

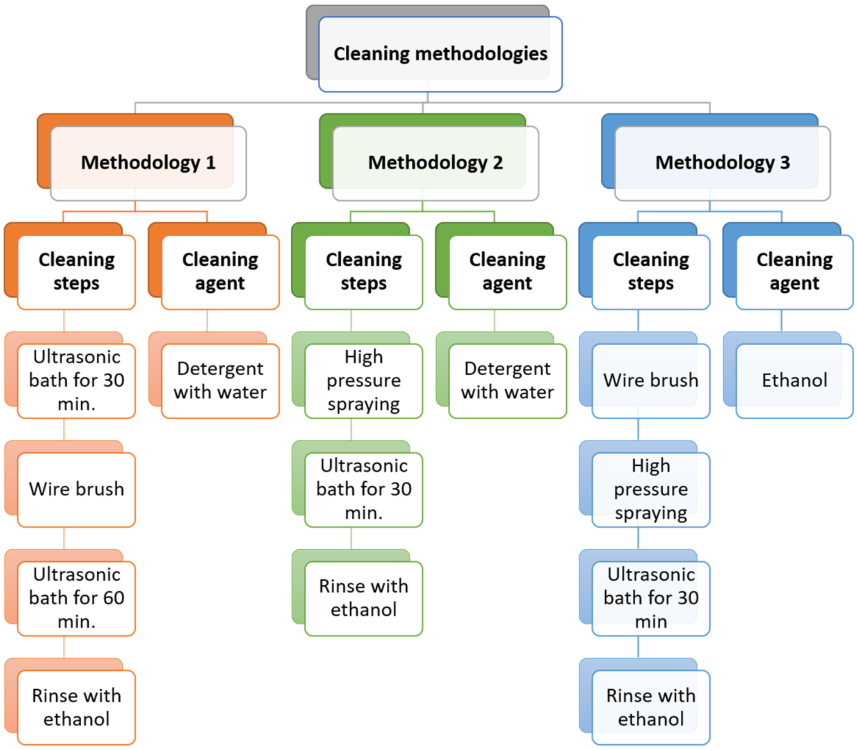

Figure 2. Methodologies used for evaluating the cleanability of the lubricants.

3. Results and Discussion

3.1. Influence of Temperature on Lubricant Degradation

The DSC curves presented in Figure 3 show that the lubricants undergo transformations as the temperature increases. Steps akin to glass transition are observed for both lubricants: at 100 °C for the polymer-based lubricant and at 70 °C for the graphite-based lubricant. At 140 °C, a peak indicating possible crystallisation of the polymer is formed, whereas the graphite-based lubricant shows multiple peaks between 120 °C and 150 °C. Above 150 °C, the graphite-based lubricant does not undergo significant changes, and in the case of the polymer-based lubricant, no changes were observed after 175 °C; at this temperature, degradation of both materials has taken place. TGA analysis shows the weight change of the lubricants as a function of temperature, where the graphite-based lubricant shows a sharp weight reduction (40%) from 100 °C to 160 °C, and the polymer-based lubricant shows an 85% weight loss from the start of the measurement until 160 °C.

The changes observed in these measurements are associated with burnout, as well as transitions related to transformations of the compounds in the lubricants. Since no detailed information on the composition is known for either lubricant, it is difficult to make an exact analysis of the different transformations that take place. However, a clear takeaway from the DSC/TGA analysis is that no significant or critical changes or degradation occurs below 100 °C for either of the lubricants. Above 100 °C, the loss of weight starts changing more rapidly, and a number of transformations start to take place before the final degradation. It is clear from this analysis that when the lubricants are exposed to temperatures above 150 °C, they will be degraded. In hot stamping of aluminium alloys, typical temperatures of the workpiece range from 300 °C to 500 °C depending on the material and the specific process parameters. On solutions where the lubricant is applied on the tool, the lubricant is expected to resist temperatures up to 400 °C for a short period of time [16]. Even if the surface temperature of the tool is not the same as the workpiece, it can still increase to levels that initiate degradation. Zheng et al. [17] calculated surface temperatures up to 180 °C during forming depending on the process parameters, such as initial die temperature, workpiece temperature, and contact pressure.

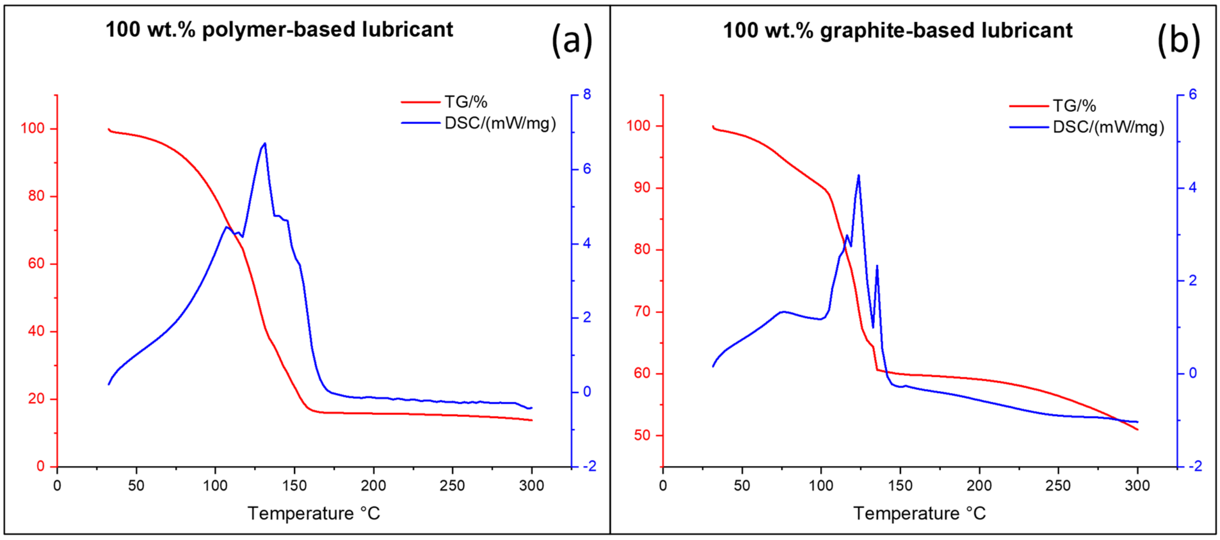

Figure 3. DSC and TGA measurements for (**a**) 100 wt.% polymer-based lubricant, and (**b**) 100 wt.% graphite-based lubricant.

Considering that the lubricant starts to degrade at 100 °C, and that the recommended usage temperature by the lubricants supplier is between 80–150 °C, the tribological tests were performed at 100 °C and at 300 °C—consequently evaluating the lubricant before any significant change has taken place—and at 300 °C, which is a relevant temperature for the hot stamping process of aluminium alloys, as well as a severe temperature for the lubricants.

For the cleanability studies, the temperatures selected were 150 °C—which is the temperature where degradation has already had a significant impact—and 300 °C, where the lubricant should be fully degraded, consequently giving direct information on how the transformations of the lubricants affect their cleanability.

3.2. Friction and Wear Behaviour

3.2.1. Influence of Temperature on Lubricant Behaviour

Figure 4 shows the friction behaviour from the tests at 100 °C and 300 °C using a 10 wt.% concentration on the lubricants. The results for graphite-based lubricant at 100 °C (Figure 4a) and 300 °C (Figure 4b) show relatively low and steady coefficients of friction (COF) over the entire duration of the test after an initially high coefficient of friction. The behaviour was more reproducible at low temperatures than at high temperatures. The higher friction observed at the beginning of the test is a common observation in this type of test; in the initial states, the friction coefficient is heavily influenced by the initial acceleration and stabilization of the test conditions. Nevertheless, it was observed that the initial coefficient of friction was higher for the tests at 300 °C than those at 100 °C. In the steady state, at 100 °C, the average COF was 0.12 ± 0.01, whereas at 300 °C, it was slightly higher and with more scatter (0.18 ± 0.02). The higher scatter was the result of the outlier results from one of the repeat tests.

In contrast, the tests performed using the polymer-based lubricant resulted in higher COF and more unstable behaviour. As shown in Figure 4d, at 100 °C, an initial friction peak was observed, followed by a reduction in the coefficient of friction; however, the behaviour was different for every test, although a dominant trend was for the friction coefficient to increase with sliding. For these tests, an average COF of 0.29 ± 0.28 was measured. For the tests at 300 °C, an even more unstable friction was obtained (Figure 4e). In this case, the average COF was 0.81 ± 0.63. Due to the high scatter at this temperature, two additional tests were performed. In this case, full failure of the lubricant was observed with the occurrence of aluminium transfer onto the tool. Examples of the tool surface after the tests at 300 °C are shown in Figure 4c,f for the graphite and the polymer-based lubricants, respectively. It can be seen that only the tests using the polymer-based lubricant underwent aluminium transfer.

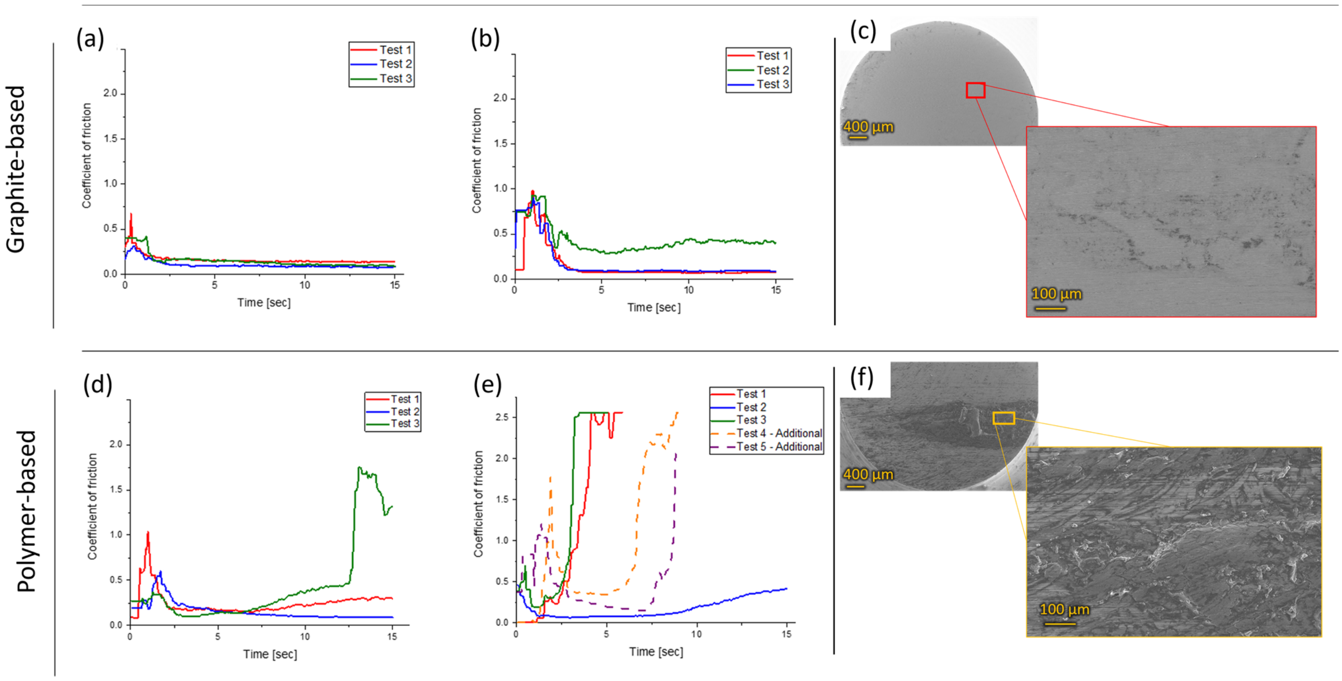

Figure 4. Coefficient of friction of the graphite-based lubricant at (**a**) 100 °C and (**b**) 300 °C, and for the polymer-based lubricant at (**d**) 100 °C and (**e**) 300 °C. Worn surfaces from tests at 300 °C are also shown: (**c**) graphite-based, (**f**) polymer-based.

The degradation of the lubricant with the increased temperature is not unexpected. It has been observed in other studies that increasing temperature results in a larger scatter and higher coefficient of friction of various lubricated systems [4,9,15].

This indicates that despite the observed changes in the DSC/TGA, the graphite-based lubricant is able to maintain good lubrication over the tested temperature range, and although the stability seems to be affected, the lubricant maintains low friction and wear protection. It also possible that the changes observed in the DSC/TGA analysis are mostly limited to the carrier, and no significant degradation or changes on the graphite occur at this temperature.

In contrast, the polymer-based lubricant was only effective in preventing wear at the recommended temperature (100 °C), but friction stability was affected. Furthermore, an increase in temperature results in actual failure of the lubricant and direct contact between the two samples.

A similar tribological response for other lubricants has been reported in the literature, both in terms of wear and friction response in simple test configurations [9,15], and even in simulative testing for hot stamping [18]. This highlights the potential of the current methodology for investigating the tribological response of new lubricants for hot stamping of aluminium.

3.2.2. Influence of Lubricant Concentration on the Tribological Behaviour

The effect of lubricant concentration was evaluated for both lubricants. In this case, tests were performed at 300 °C. This was conducted with the aim of assessing whether the lubricants are able to provide sufficient lubrication at the harsher condition by increasing the lubricant concentration. Figure 5 shows the comparison for the two lubricants with 10, 50, and 100 wt.% concentrations. The behaviour of the graphite-based lubricant did not change significantly when increasing the concentration. A slight increase in friction with the increase in concentration was observed; for the 10 and 50 wt.%, similar average values were seen for both concentrations 0.18 ± 0.2 and 0.16 ± 0.2, respectively. Further increasing the concentration to 100 wt.% showed a slight increase to an average COF up to 0.23 ± 0.11.

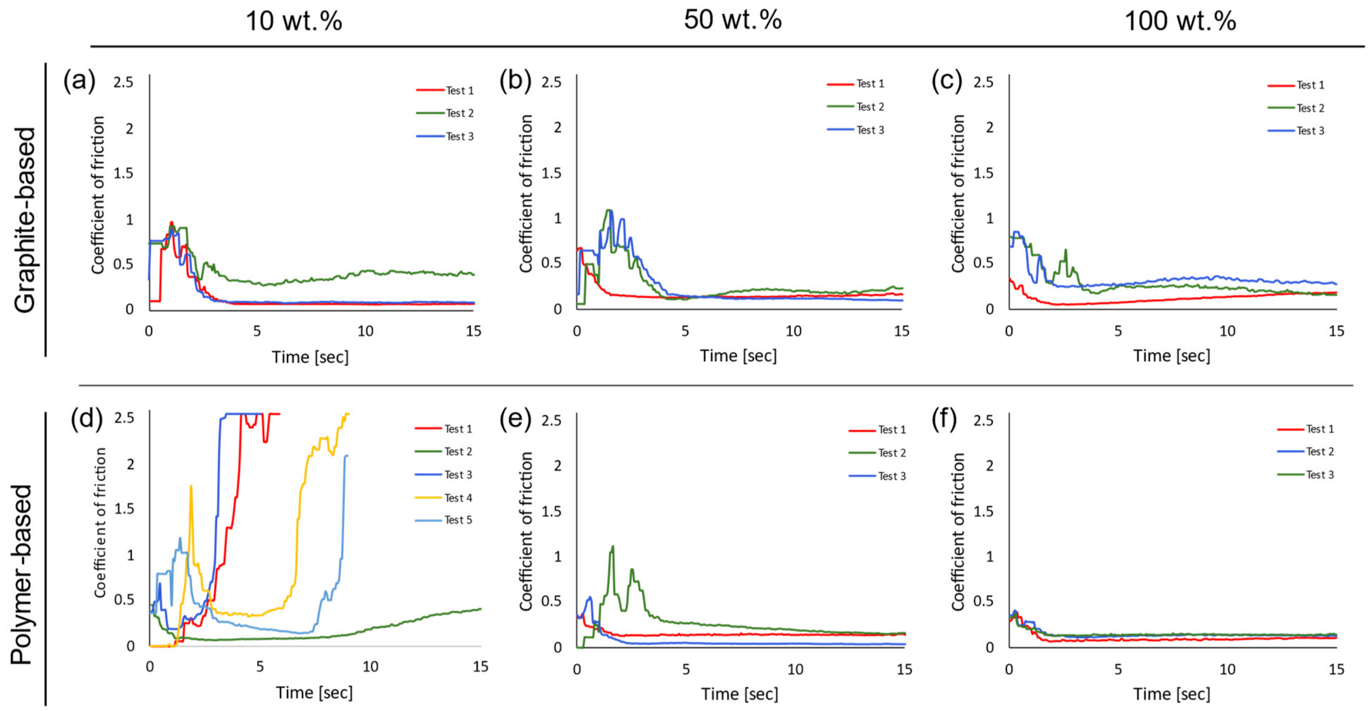

Figure 5. Coefficient of friction of repeat tests from the graphite-based lubricant with (**a**) 10 wt.%, (**b**) 50 wt.%, (**c**) 100 wt.% concentration; and from the polymer-based lubricant with (**d**) 10 wt.%, (**e**) 50 wt.%, (**f**) 100 wt.% concentration. All tests were conducted at 300 °C.

In contrast, the polymeric lubricant showed a significant improvement in terms of repeatability, stability, and lower friction with the increase in concentration. In this case, 50 wt.% and 100 wt.% did not undergo failure of the lubricant. Furthermore, the undiluted lubricant showed the most stable and lowest friction of all tested conditions, with an average CoF of 0.12 ± 0.01 at 100 wt.% compared to 0.14 ± 0.2 at 50 wt.%.

The worn surfaces after the tribological tests for the 50 and 100 wt.% are shown in Figure 6. No significant wear took place on the tool pin specimens for either lubricant. However, lubricant remnants were observed in all tests, with the graphite-based lubricant leaving significantly more residue after the tests when it was undiluted.

In contrast, the aluminium alloy showed signs of wear for both lubricants when a concentration of 50 wt.% was used. Interestingly, even though wear did take place on the aluminium alloy specimen, no significant material transfer nor sudden increases on friction were seen for any of these tests. It is unclear if the damage observed in the aluminium is generated during the first few seconds of the tests, or later during the test. What is apparent is that regardless of when the damage is generated, the lubricants are able to prevent material transfer on the tool steel. It has been suggested by Podgordnik et al. [19] that an optimal concentration for solid lubrication exists, increasing concentration and lubricant size results in a reduction of the coefficient of friction and wear only up to a certain point. It is likely that in this study, the optimal lubricant concentration was not observed, but it is noteworthy that the test methodology is able to differentiate the influence of this parameter.

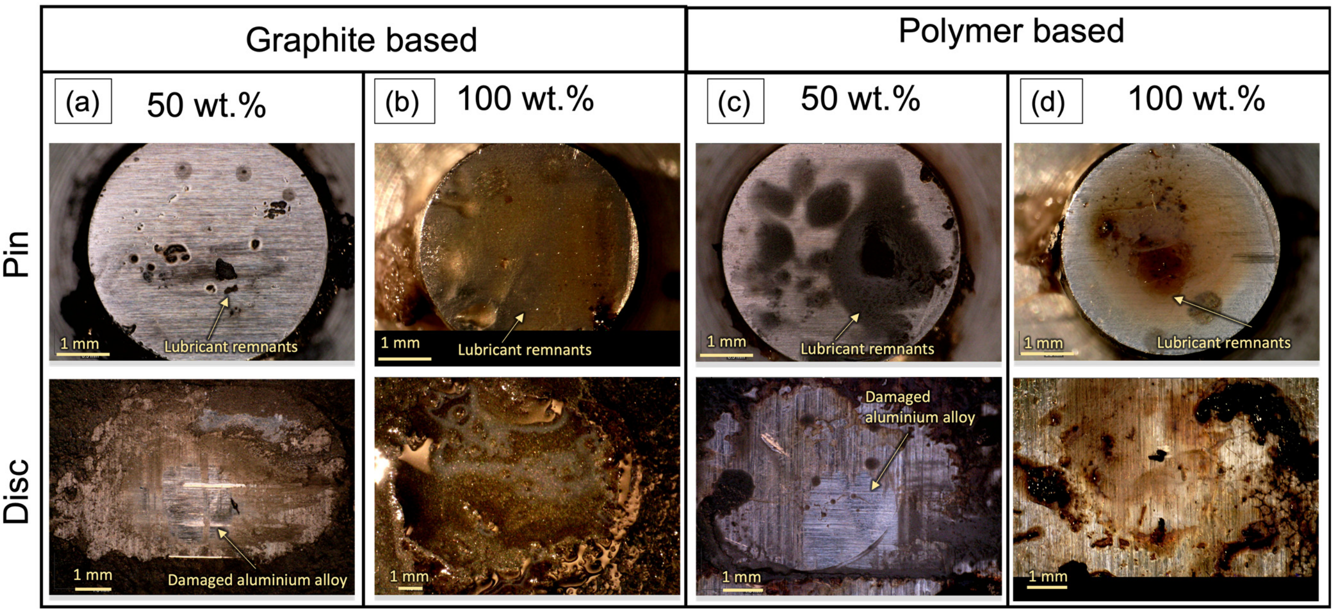

Figure 6. Representative worn surfaces of the tool pin and the aluminium plate after the tribological tests for the graphite-based lubricant with (**a**) 50 wt.% and (**b**) 100 wt.%, and the polymer-based lubricant with (**c**) 50 wt.% and (**d**) 100 wt.%. Sliding left to right.

A more in-depth analysis of the worn tool steel specimens using the undiluted lubricants was conducted by means of SEM (Figure 7). The specimen where graphite-based lubricant was used showed no signs of surface damage in the form of adhesive or abrasive wear on the tool. In contrast, when the polymer-based lubricant was used, localized abrasive wear was seen, and transferred aluminium was detected, as highlighted by the EDS analysis, even though it was difficult to see. It is likely that the transferred aluminium is in a thin layer that is transparent to the secondary electrons. It is important to note that friction for this test was the most stable, lowest, and most repeatable. As mentioned earlier, it is possible that the damage observed in this case takes place during the initial stages of the test. The specimens are initially statically loaded, and then accelerated to reach the set sliding conditions; it is likely that the damage observed occurs during the acceleration stage, as harsher conditions are experienced in the contact. It is also important to mention that this was seen only on one specimen, which indicates that wear is mostly negligible with this lubricant under the tested conditions.

Graphite-based lubricants have been reported to operate well in hot forming operation or at high temperatures. Some other studies on polymeric lubricants have also highlighted the possibility of utilising them in hot stamping conditions [18,20,21]. This study highlights the importance of finding the best concentration for the lubricant, as well as controlling the temperature.

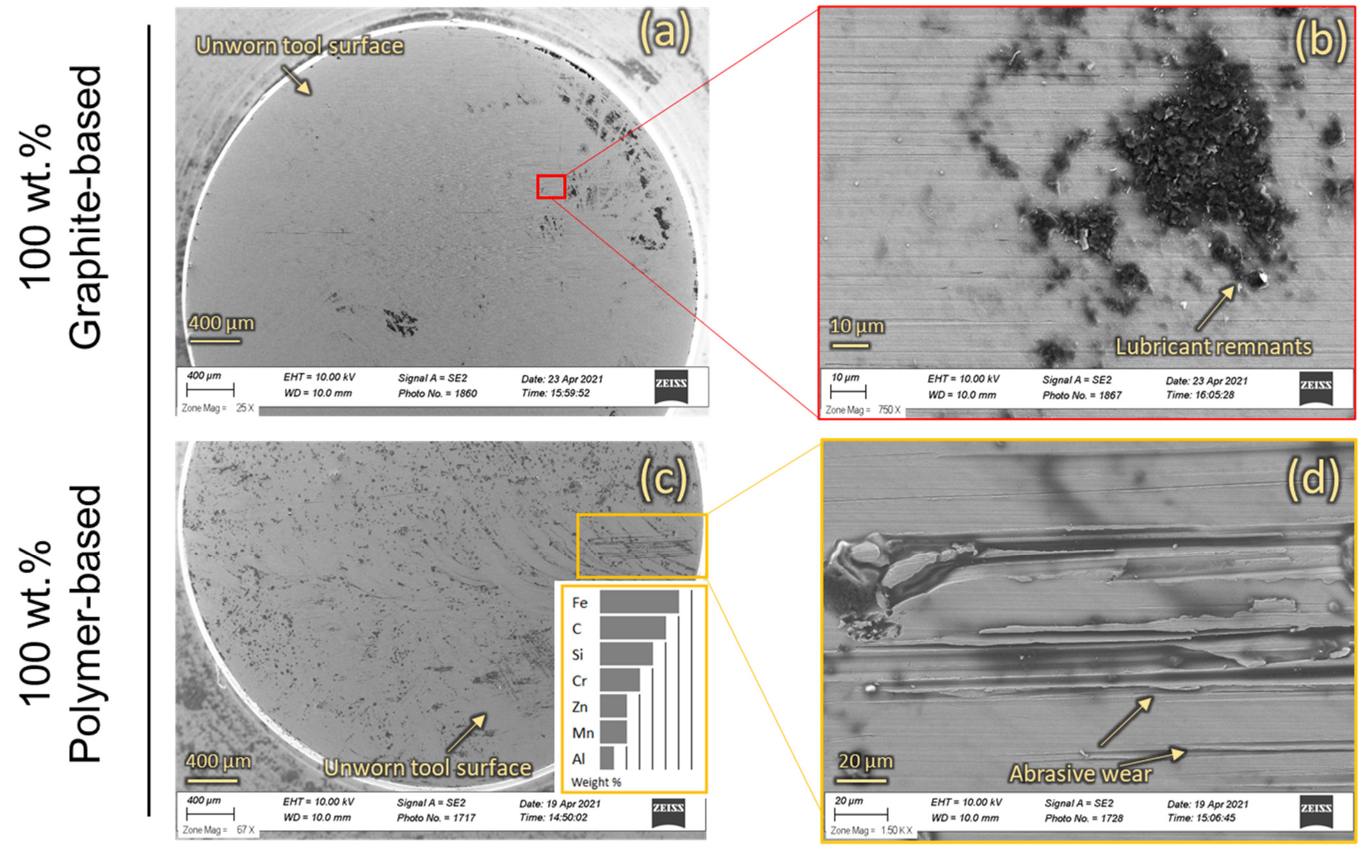

Figure 7. SEM micrographs of the worn tool steel surface observed with 100 wt.% graphite-based lubricant (**a**) overview, (**b**) detail of lubricant accumulation; and with 100 wt.% polymer-based lubricant (**c**) overview, (**d**) detail of worn surface (EDS corresponds to area in (**c**)). Observation done with 10 kV and 10 mm working distance.

3.3. Lubricant Cleanability Study

Figure 8 shows the results after the cleanability tests. It was observed that at 150 °C, the lubricants could be cleaned by all of the different methodologies. However, once exposed to 300 °C, they cannot be cleaned effectively by any of the cleaning methods. Use of ethanol resulted in more effective cleaning of the surfaces, particularly for the polymer-based lubricant, and even after exposure to high temperatures, some of the lubricant was removed. These results show that the changes that the lubricants undergo after exposure to high temperature result in a strong bonding to the surfaces. Even if the lubricants manage to maintain a certain degree of lubrication, such as the case of the graphite-based lubricant, the end result is that a more aggressive cleaning procedure needs to be used in order to clean the surfaces.

The specimens cleaned at 150 °C were further analysed at higher magnification with SEM (Figure 9). In these images, the darker contrast is caused by remnants of the lubricant after the different cleaning methodologies. It is clearer from these images that ethanol is the best agent to effectively remove the lubricant, even if it may visually seem that the surfaces are clean using the other methodologies. This is more prominent for the case of the polymeric lubricant, where the surface is essentially cleaned completely after cleaning with ethanol (Figure 9c). The graphitic lubricant left remnants after all of the methodologies, but attained a significantly clean surface with all the different cleaning methodologies.

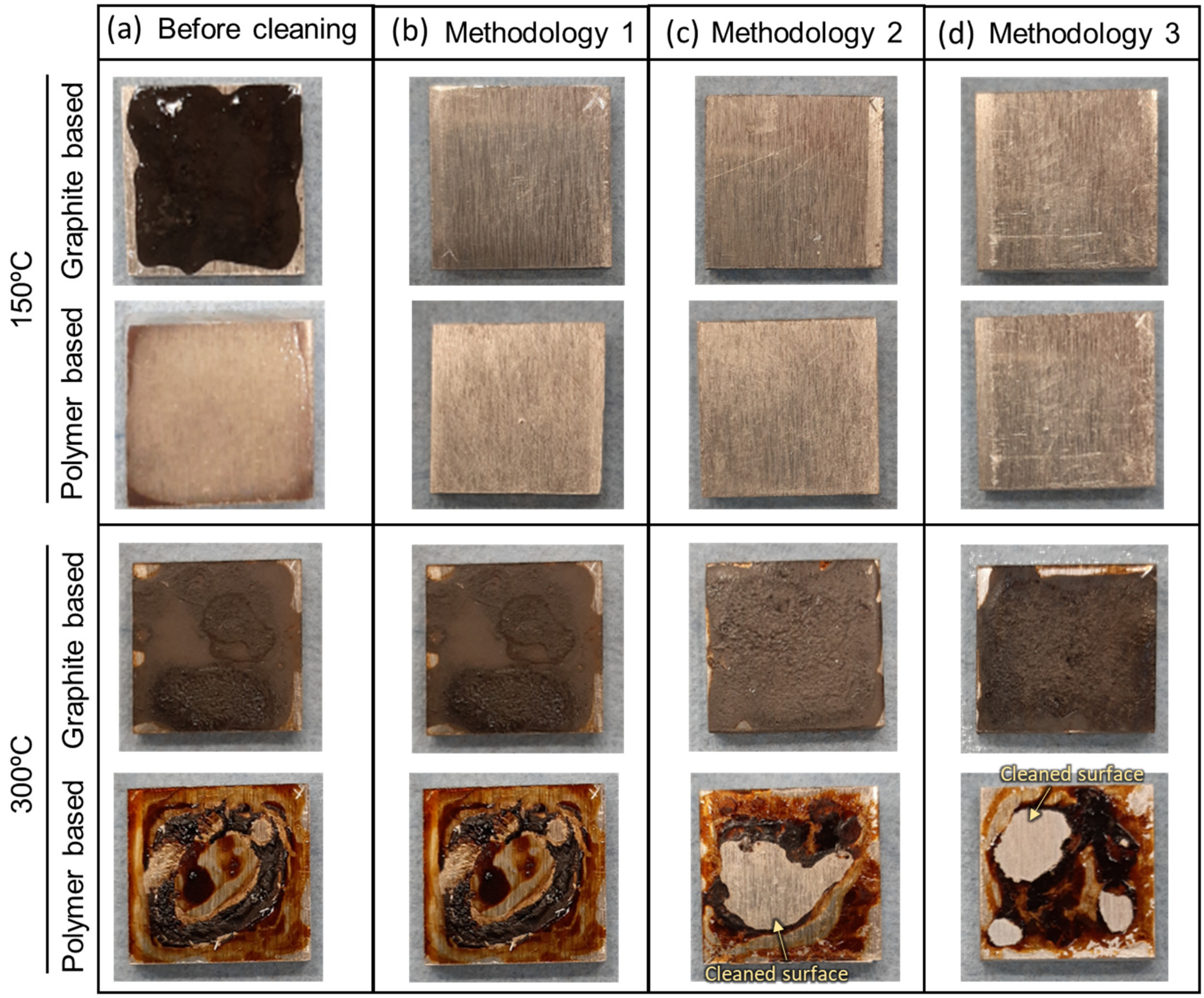

Figure 8. Photographs of the aluminium surfaces after the different cleaning methodologies at 150 °C and 300 °C for both lubricants. (**a**) before testing, (**b**) after methodology 1, (**c**) after methodology 2, and (**d**) after methodology 3.

3.4. Test Method Discussion

This test methodology showcased that both lubricants operate well at lower temperatures, but relatively worse behaviour exists for the polymeric lubricant, which worsens further as the temperature increases. The test methodology was also able to showcase the effect of the lubricant concentration, where it was observed that with the right concentration, the polymeric lubricant could maintain low friction and wear. The tests also highlighted the direct effects of temperature and lubricant on cleanability. It is clear that further analysis is necessary on this topic, but this study shows that simple tests can be performed to further improve the knowledge in this regard.

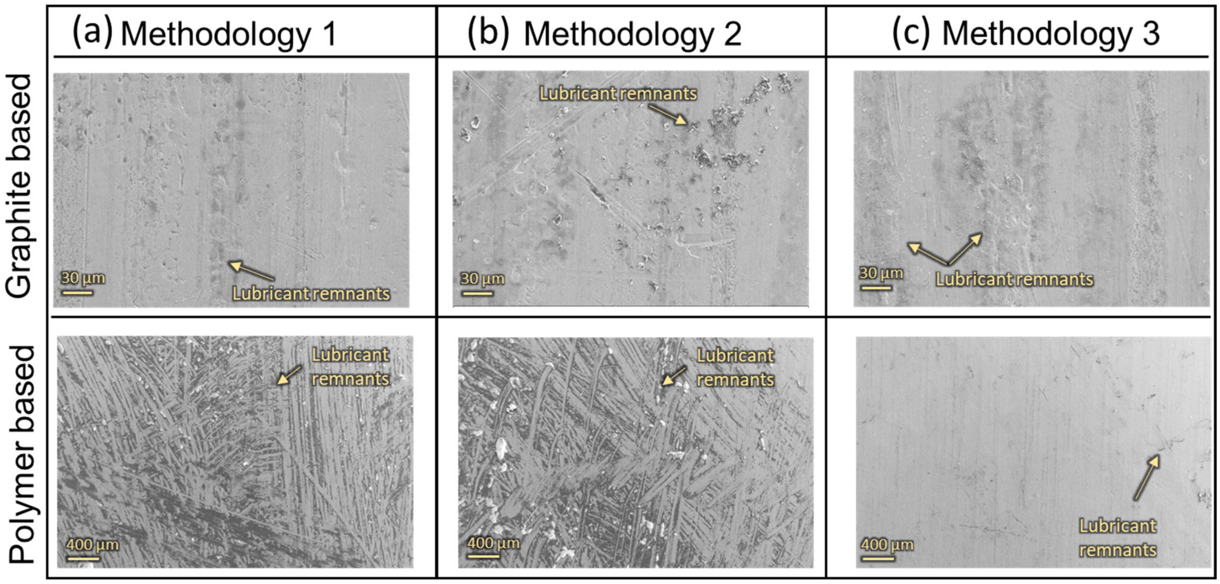

Figure 9. SEM micrographs of the aluminium surfaces after the different cleaning methodologies at 150 °C for both lubricants. (**a**) after methodology 1, (**b**) after methodology 2, and (**c**) after methodology 3. Observation done with 10 kV and 10 mm working distance.

4. Conclusions

In this study, a test methodology to evaluate the tribological behaviour of lubricants intended for hot stamping of aluminium was developed. Two lubricants were evaluated with this methodology and their cleanability was also assessed. The salient conclusions from this study are as follows:

- The developed test methodology to evaluate lubricants at high temperatures proved to be effective. The tests showed good repeatability and differences in the friction, and wear behaviour of the lubricants could be assessed.
- An increase in friction and frictional instabilities as temperature increases due to changes and/or degradation that the lubricants undergo. For graphite-based lubricant, the CoF increased from 0.12 to 0.18, and for the polymer-based lubricant, it increased from 0.29 to 0.81 when their concentration was 10 wt.%.
- The graphite-based lubricant results in a higher coefficient of friction when increasing concentration from 0.18 to 0.23 at 300 °C, but is able to prevent galling on the tool steel.
- The polymeric lubricant is more effective at higher concentrations, reducing the CoF down to 0.12 in its undiluted form.
- The best cleanability was achieved when using ethanol, particularly for the polymer-based lubricant.
- The cleanability of the lubricants significantly worsens as a result of the changes that the lubricants undergo when exposed to higher temperatures.

The results have highlighted the potential of the present methodology to evaluate and rank lubricant performance and their cleanability within the context of hot stamping of aluminium. It is also important to note that the method can be improved to account for other parameters such as contact pressure, and further improvements need to be addressed to more quantifiably rank the cleanability of the lubricants.

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