

Development of a new green industrial lubricant for stamping operations – Application to Stainless Steels

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Abstract. Thanks to a fruitful collaboration between different academic and industrial research entities, a new green lubricant solution AFULudine has been developed for stamping operations in substitution to the usual mineral oils, with the idea and the wish to combine different essential properties such as a low environmental impact and ease-of-use according to the market needs and the more and more restrictive environmental legislation (e.g. REACH). Based on the Self-Assembled-Monolayer (SAM) principle and so, on a perfect control of the chemical reaction between the solution and the substrate (grafting of molecules onto the surface), AFULudine offers an efficient technical answer for improving stamping processes. The present study, focused on stainless steel stampings (comparison between a 441-1.4509 grade and a 304-1.4301 grade), investigates the performances of this new green industrial lubricant at different levels: from laboratory tests to industrial conditions through the production of hundreds of parts. Additional results coming from different tribometers will allow us to appreciate and retrieve more local information about the tribofilm creation during sliding. Moreover, the comparison with different mineral oils currently used at industrial scale, will strengthen the AFULudine performances. Indeed, this new solution usually outperforms a majority of such oils whatever their viscosity and their own composition: formulation, content of extreme pressure additives (Cl, S, P)...

1. Introduction

Lubrication of stamping operations is today widely assumed as absolutely necessary to allow part feasibility and preserve surface integrity of both stamped parts and tools. This finding appears to be particularly important concerning the forming operations of stainless steel grades, whatever the mechanical properties, for which well-known issues of adhesion or transfer of work material to the tool can easily occur if no precautions are taken [1, 2]. The figure 1 illustrates the current lubrication offer dedicated to stainless steels. Mainly based on petrochemical processes and widely dominated by few industrial companies, a compromise between performance, correlated to friction coefficient and easy-of-use, linked to the viscosity, must be done. Today, no concrete alternative solutions exist to substitute the most efficient lubricants, containing additives such as sulphur, phosphorous or chlorine. These compounds are designed to chemically react with the metal surface during mechanical solicitations, forming sheared layers of sulfides, phosphides and chlorides according to the induced working temperatures [3]. In fact, the higher efficiency of the chlorine additive with stainless steels is

due to a good adequacy between the working temperatures inherent to drawing processes and its activation temperature.

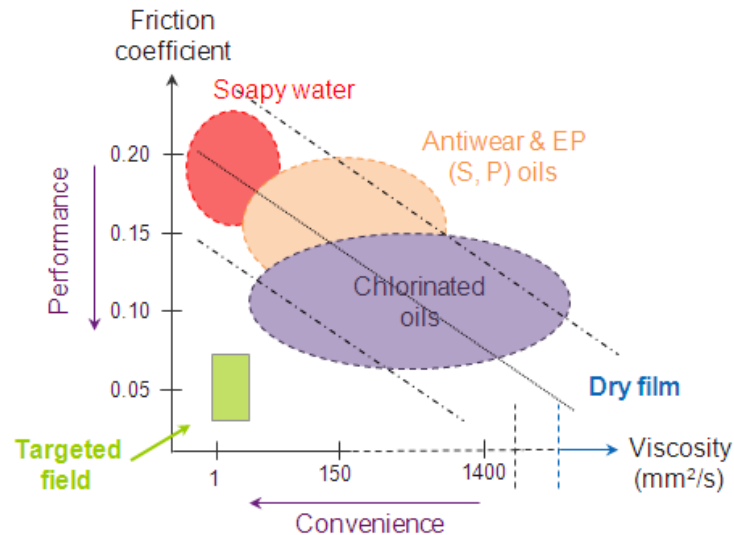


Figure 1. Schematic view of the current offer in terms of lubricants dedicated to stainless steels – a daily compromise has to be done between convenience (easy-of-use) and product performance. The AFULudine target is highlighted by the green box.

The development of the AFULudine solution is based on the wish to perfectly control the chemical reaction between specific active molecules and metallic oxidized substrates such as stainless steels. With the idea to voluntarily control chemical reactions instead of hoping that these ones will occur during sliding, the developed solution ensures surface functionalisation by using organic molecular assemblies like Ulman and al. previously did [4], and more especially by using alkylphosphonic acid molecules. These molecules are well-known to act as a protective layers formed by chemical reaction with the metal surface due to a fairly high level of chemical activity [5]. Raman and al [6] studied their ability to create a Self-Assembled Monolayer (SAM) on the surface of an oxidized austenitic stainless steel (316L). In more recent studies [7, 8, 9, 10], significant improvement of the tribological behaviour of different substrates (copper, titanium, stainless steels) was demonstrated and assigned to alkylphosphonic acids molecules used as nano-size liquid additives in lubricant, highlighting more especially the formation of low-friction tribofilm during sliding, responsible for the surface protection. Such active molecules are the very essence of the AFULudine solution, which constitutes today a more environmentally friendly lubrication solution than the standard petrochemical lubricants.

The present study investigates at different experimental scales until industrial production the full potential of the AFULudine solution for two different stainless steel grades: a 1.4301-189ED austenitic grade and a 1.4509-K41 ferritic grade, according to mechanical solicitations representative of deep drawing operations. The lubrication strength for such shrink drawing deformation path is widely validated today. Verma and al. [12] concluded for example that the limiting drawing ratio (LDR) decreasing with increase in friction. In addition to the performance comparison with some industrial mineral oils dedicated to stainless steels, the influence of the AFULudine processing conditions (solution concentrations + lubrication process) on the product performance will be highlighted. Direct correlations between a tribometer especially designed by Roizard and al. [11] to be highly representative of stamping operations [11] and two stamping presses (a laboratory press and a single effect production press) will be furthermore established.

2. Experimental

2.1. Material

Several industrial austenitic and ferritic stainless steel grades were used as received with different finish surface states: 2R (bright annealed surface) or 2B (surface after a pickling step followed by a Skin-Pass operation), without any additional preparation steps whatever the mechanical characterizations done. The intrinsic roughness variations between these different surface states will not be taken into account in the present study. Recent works on ferritic stainless steels [9] showed a limited contribution of the roughness in the tribofilm formation. The table 1 sums up the standard chemical compositions of these grades as well as their main tensile properties.

Table 1. Chemical composition of the Stainless Steel grades

Grade (EN-ASTM-Commercial codes)	C	Si	Mn	Cr	Ni	Others
1.4509 – 441 – K41	0.015	0.60	0.30	17.80	-	Ti+Nb=0.65
1.4301 – 304 – 189ED	0.040	0.40	1.20	18.20	8.10	-
1.4301 – 304 – 189DDQ	0.045	0.40	1.10	18.20	9.10	-

Table 2. Tensile properties of the Stainless Steel grades

Grade (EN-ASTM-Commercial codes)	Thickness (mm)	UTS (MPa)	YS (MPa)	A%	r_N
1.4509 – 441 – K41	1	480	310	30	1,285
1.4301 – 304 – 189ED	1	630	285	57	0,970
1.4301 – 304 – 189DDQ	0,8	610	270	57	0,936

UTS = Ultimate Tensile Stress, YS = Yield Strength, A% = Maximal elongation, r_N = Normal anisotropy

2.2. AFULudine solution & other mineral oils

Based on the use of alkylphosphonic acids as organic friction modifiers, the AFULudine solution has been thought to ensure an industrial easy of use thanks to a hydro-alcoholic solvent. Several AFULudine solutions were investigated in the present study all based on a single carbon chain length. These ones will not be fully detailed in the present paper in terms of composition and process in order to preserve as much as possible the internal know-how. They will be identified mainly thanks to their relative concentration compared to a normalized reference: from C1 (2%) to C9 (300%), with C7 (100%) defined as the current reference. Likewise, the different industrial processes used to lubricate the substrates will not be fully described either, but simply identified by the following notation: from P1 to P5. The contact time with the surface (decreasing from P1 to P4, leading to a productivity improvement), the quantity of lubricant and the type of lubrication system are the changing parameters between the different processes, which can be after all considered as perfectly adapted to standard industrial production equipments (except the P5 process corresponding to a specific lab treatment).

Other industrial lubricants from the petrochemical industry were characterized on this study and confronted to the AFULudine solution. Highly representative of the market, more than a dozen of the mineral oils, containing different chemical additives such as extreme-pressure (S, P, Cl) or other anti-wear elements were tested. The different oils will only be distinguished from one to another by their respective Chlorine content: with or without Cl. No distinction in terms of viscosity will be done. The grammage was maintained quite constant, between 5 and 10g/m², whatever the lubricant. Notice that the oil number attributed on a graph will not be necessarily the same on another graph.

2.3. Tribological measurements & Stamping characterizations

The AFULudine performances were tested and quantified at different scales: from lab tests, with two different tribometers and one mechanical Erichsen stamping press, to industrial equipments dedicated amongst others to the daily production of pans (Cristel[®]). We made a point of choosing experimental laboratory tests consistent with the friction conditions encountered during stamping operations (cf. Introduction). The figures 2 and 3 illustrate this multi-scale characterization approach with, on the left side (Figure 2) the synoptic description of the strip drawing tribometer used (with a cylinder/cylinder contact) and, on the right side (Figure 3) the schematic view of a single effect hydraulic press as used by Cristel [13].

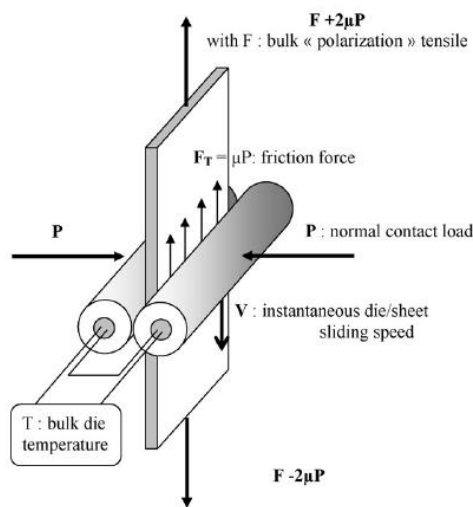


Figure 2. Synoptic description of strip drawing tribometer – Lab scale [11]

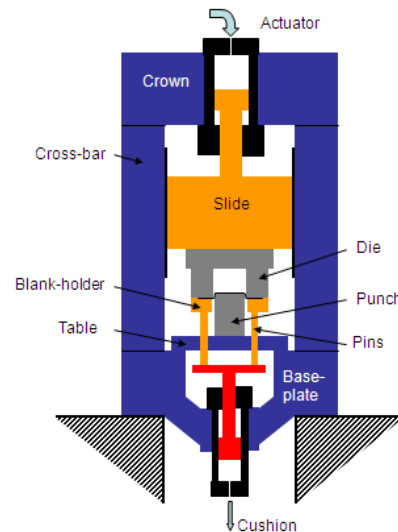


Figure 3. Schematic view of a single effect press [13]

Representative of contact conditions under the blank-holder and the die, the originality of the strip drawing tribometer compared to other equivalent tribometers with different tool geometries (e.g. flat/flat used by Kim and al. [14]) is to discretize the friction thanks to the cylinder/plan configuration. This allows to perform multi-pass tests in order to accurately quantify the friction evolution according to the tool path seen by the sheet and the setting parameters used. In this study, results will mainly integrate data coming from tests carried out at room temperature changing only the type of lubricant, without plastic deformation of the sheet. Experimental settings are summed up in the Table 2.

To complete this first analysis scale, a rotational pin-on disk tribometer (with a circular track of 3,5 mm radius) has been used for quantifying the intrinsic tribofilm resistance [7, 9] and its ability to support very long sollicitation distance according to the experimental settings used (Table 2).

As a complement to these tribological data, stamping tests were carried out with an Erichsen press (20T capacity – 142.20 model), with the wish once again to quantify the intrinsic impact of the lubricant on the Limit Drawing Ratio (LDR). Indeed, when changing only the lubricant, LDR value is directly correlated to the lubricant performance due to the different friction phenomena inherent to the forming operation. Each characterization has been performed at room temperature, with equivalent experimental settings (Table 2) and replicated 3 times to ensure the repeatability. With a blank-holder pressure equal to 6kN, the experimental parameters have been optimally chosen regarding the deep drawing field of each grade/thickness couple. Moreover, for each test, a recording system allowed us to measure the drawing force applied during to the punch stroke.

To finally validate the AFULudine performance at an industrial scale, collaboration was set up with a faithful customer of Aperam: Cristel, producer of high-end cooking utensils, which currently use an efficient chlorinated mineral oil for its stamping operations. Different production campaigns were carried out for positioning the AFULudine performance in series production: small batches of 20 pans ($\varnothing 240\text{mm}$) were firstly done for studying the impact of the surface preparation (P1, P2, P3 and P4) before opting for a 200 parts set in order to integrate potential mass production effects (production rate: 4parts/min). All the tests were conducted with a constant C1 concentration on a single effect press as illustrated by the figure 3, with instrumented tools continuously recording for example the maximal drawing force applied by the slide during the punch stroke. As a result of the device conception, the only measurement of the drawing force applied by the slide informs directly of the lubrication contribution to the forming operation, provided that other parameters (material, blank-holder force, tools kinematic) remain unchanged.

The table 2 finally synthesizes the experimental devices and conditions used in this study to compare the AFULudine solution with the different industrial oils.

Table 2. Experimental settings used with the different equipments.

Equipment	Experimental settings	Substrate
Multi-pass strip drawing tribometer	V = 10mm/min – F = 4kN (Hertzian mean contact pressure = 420MPa) – Sliding distance = 10mm – 20 passes Contact surface = 50mm Z160CDV12 quenched tools ($\varnothing 10\text{mm}$)	K41 (1mm)
Rotational pin-on-disk tribometer	V = 770mm/min – F = 5N (Hertzian mean contact pressure = 860MPa) – Sliding distance = 22mm Steel ball 100C6 ($\varnothing 5\text{mm}$)	K41 (1mm)
Erichsen Press (20T)	V = 20mm/min – BH Force = 6kN – \varnothing Punch = 33mm Z160CDV12 quenched tools \varnothing variation between 2 successive discs = 2mm	K41 & 189ED (1mm)
Industrial Press (500T)	V = 2880mm/min – BH Pressure = 5.5MPa \varnothing Punch = 214mm – TiCN coating - \varnothing discs = 415mm ($\beta=1.73$)	189DDQ (0.8mm)

3. Results and discussion

3.1. Lab. tests

3.1.1. Influence of the concentration

Before comparing the AFULudine solution with other current mineral oils, the optimal working concentration of alkylphosphonic acid molecules has been firstly defined thanks to the LDR determination of a K41 1mm thick grade (Figure 4). In this figure, concentration variations are reminded by the different blue points. Instead of one specific optimal concentration, the existence of a large concentration range is highlighted, between C4 (40%) and C7 (100%) within which maximal LDR is reached (2.24 ± 0.01). Out of this range, the performance is drastically decreasing, more especially for lower concentrations for which a galling phenomenon can also be observed (C1 concentration). For higher concentration levels, the performance decrease is less critical with a ceiling value around 2.21 (± 0.01), apparently constant. This performance deterioration has no evident cause but must be due to variation in terms of surface functionalisation and/or tribofilm formation. Although the LDR values are the same for the concentrations from C4 to C7, a ranking based on the maximal force applied by the punch can be established (orange points). Thus, for the same deep drawing operation, C7 concentration induces a reduction of 2,8% of the maximal force required to perform part stamping compared to C4 concentration. More broadly, a decrease of the maximal punch force appears to be directly correlated to the increase of concentration.

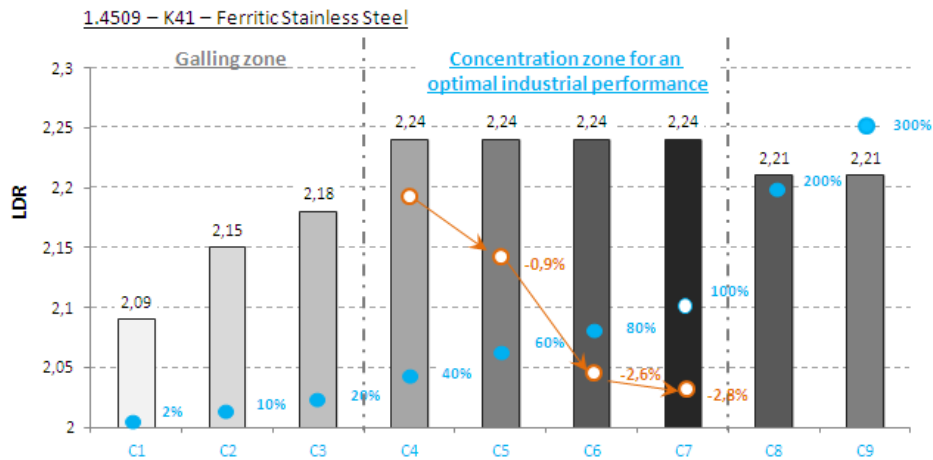


Figure 4. LDR evolution according to the AFULudine concentration: from C1 (2%) to C9 (300%) with C7 defined as reference value (100%) – Samples have been prepared thanks to the P1 process.

One of the reasons which could explain this concentration influence concerns the efficiency and the resistance of the induced tribofilm during the stamping operation according to the friction solicitations. Roizard and al [9] has recently studied the intrinsic mechanisms relative to the formation of an efficient low-friction tribofilm on the surface of a ferritic stainless steel (1.4509). A healing effect was thus highlighted and mainly correlated to the ability for new active molecules to graft at the metal surface in order to continuously replenish the tribofilm. Without internal source in terms of new molecules, a constant but irremediable deterioration of the friction coefficient has been observed until galling occurred. According to these findings, even if the test conditions were different compared to the present study, the tribofilm resistance will be defined by the quantity of active molecules available which depends on the initial concentration and the process application. These two parameters are responsible for a grafted layer having its intrinsic properties.

The figure 5 explores the friction response of samples prepared with the same AFULudine solutions than previously studied, with variations in terms of concentration between C1 and C7 (P1 preparation). Since the early stages of the test, variable performance of the tribofilm can be noticed, depending on the concentration levels in correlation with the previous LDR results. Indeed, the lower concentration, the higher friction response, even if the values are quite acceptable, between 0.08 and 0.13. Nevertheless, significant differences are quickly observed with a constant and chaotic increase of the friction coefficient for the lower C1 concentration, finally leading to a galling phenomenon. SEM views of the figure 6 illustrate the track topology after 200 cycles performed with the C1 solution (yellow point of the figure 5), bringing to light a significant degradation of the surface state. The complete absence of phosphorous on the surface after sliding as revealed by the EDS analysis strengthens the hypothesis of a complete deterioration of the tribofilm during sliding.

Increasing the concentration level of active molecules is finally leading to improve the low-friction tribofilm efficiency and its time stability until galling. A good correlation exists between this results and the ranking presented in figure 4 in terms of LDR for equivalent concentrations, mainly explained by the intrinsic tribofilm formation whatever the mechanical solicitations.

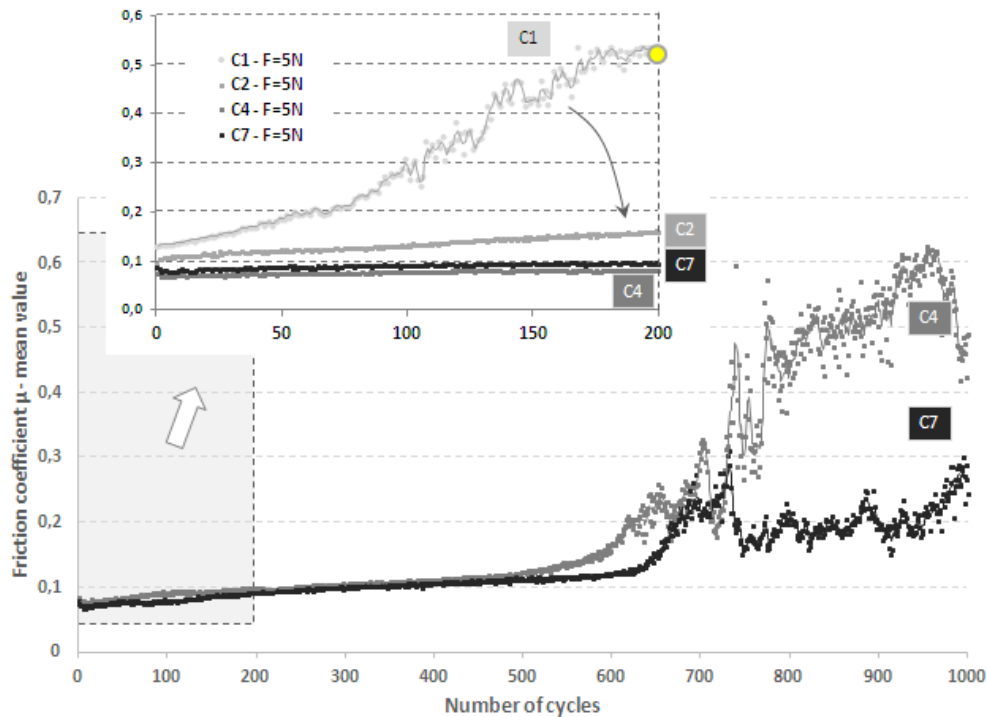


Figure 5. Evolution of mean friction coefficient versus number of cycles (Rotational pin on disk tribometer) for four different AFULudine concentrations: C1, C2, C4 and C7, from light to dark curves respectively. Samples have been prepared thanks to the P1 process

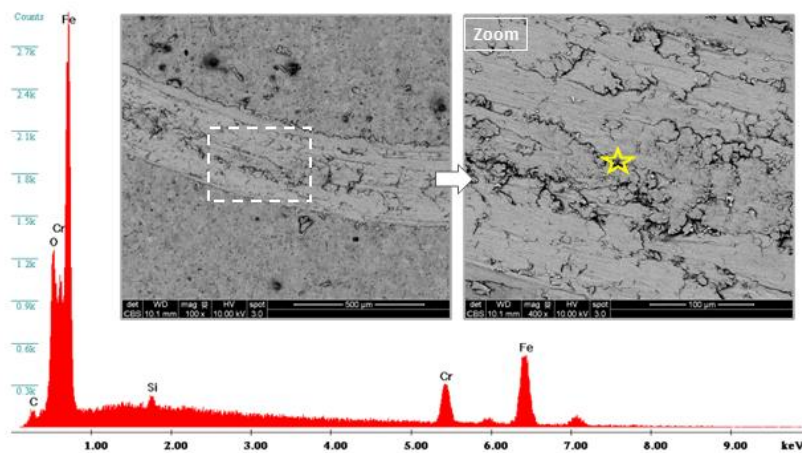


Figure 6. SEM images (back scattered electrons) of the track after sliding test on rotational pin on disk tribometer where 200 cycles were performed with the C1 AFULudine solution, prepared thanks to the P1 process. EDS analysis was performed on the yellow star location.

3.1.2. Comparison with other industrial lubricants

The figure 7 compares LDR values obtained for more than a dozen of industrial lubricants, only distinguished from one to another by their chlorine content, with the AFULudine performance at the optimal concentration (C7). A laboratory grease (based on graphite) and a new non-oil lubricant suggested as a green alternative to mineral oils were also tested and compared. The exercise was done for a ferritic (1.4509-K41) and an austenitic (1.4301-189ED) stainless steels, presented in figure 7 a) and b) respectively.

Even if the ferritic stainless steel grade highlights on average logically higher LDR values than with the austenitic grade, thanks to a direct effect of the material anisotropy, it is interesting to remark an equivalent ranking of the different lubricants according their chlorine content: higher drawability is allowed by using this extreme pressure additive, whatever the oil viscosity and the substrate nature. It is also interesting to underline the remarkable performance of the AFULudine solution, at least equivalent or even better than a majority of chlorinated oils. Its performance even matches with the laboratory grease one for the 1.4301-189ED austenitic grade. Unlike the direct competitor non-oil lubricant which appears to be sensitive to the substrate nature, the operating principle of AFULudine is relevant for all the stainless steels grades. Moreover, the scope of progress is still great as can be seen on the figure 7 a) with a 2.35 LDR value reached for the AFULudine solution prepared in laboratory conditions (C7-P5). New developments are currently engaged to achieve equivalent performance in industrial conditions. It may also be noticed that oil 2 used with both stainless steel grades refers to the chlorinated lubricant daily used by Cristel. With LDR values equal to 2.25 (1.4509-K41) and 2.11 (1.4301-189ED), this one can be considered as appropriate.

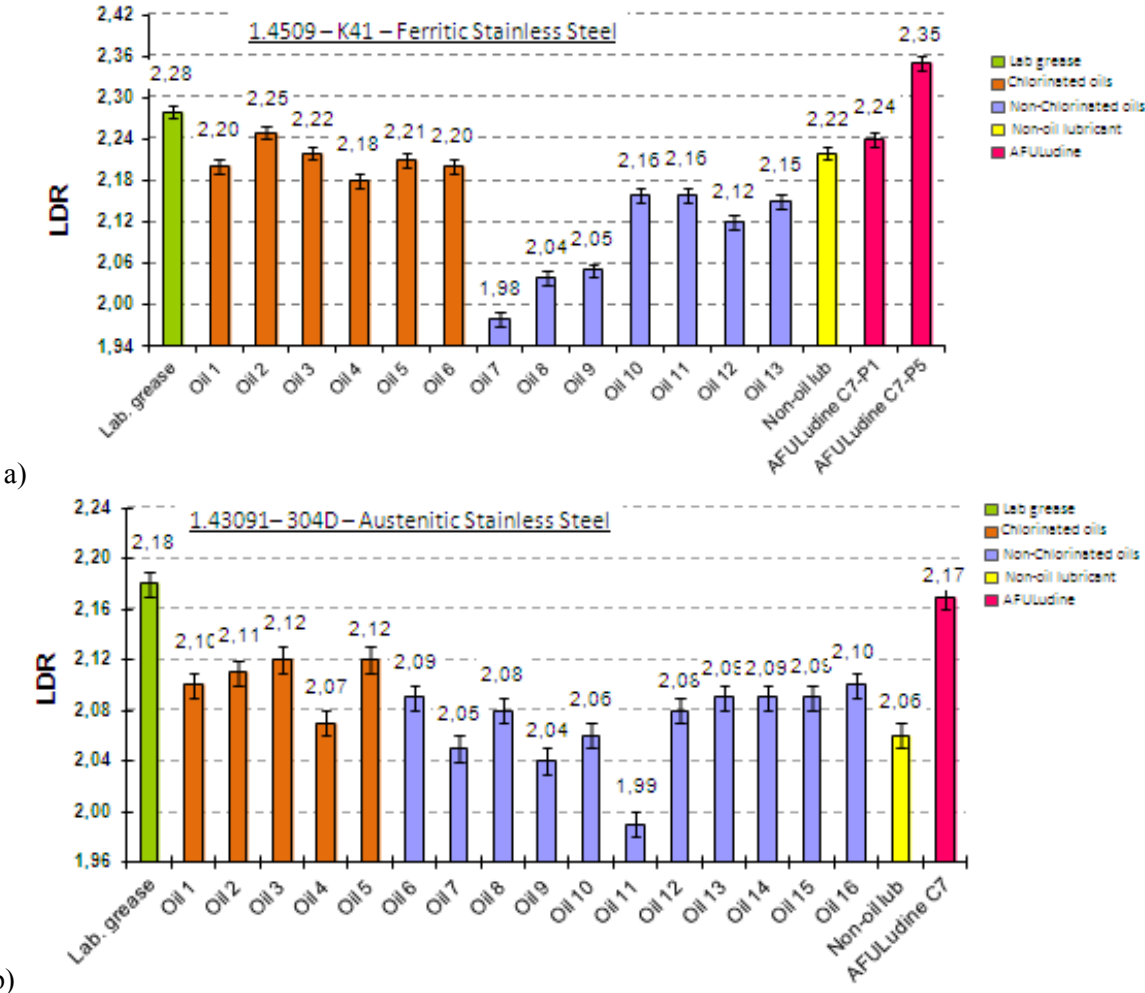


Figure 7. LDR comparison between different industrial oils with (orange bars) or without (blue bars) chlorine additive, a lab grease (green bars), a non-oil industrial lubricant (yellow bars) and the AFULudine solution (pink bars - C7 concentration – P1 preparation) for a) a 1.4509 ferritic stainless steel and b) a 1.4301 austenitic stainless steel. Notice that for the ferritic grade, the specific laboratory preparation P5 of the AFULudine solution (C7) was also tested.

The figure 8 provides another point of view of the AFULudine performance through experimental data obtained on the multi-pass strip drawing tribometer on a 1.4509-K41 grade. In the interests of clarity, just few different mineral oils were compared with AFULudine (C7-P1 and C7-P5). With a friction coefficient close to 0.04 during the 20 passes of the test (on the same track), AFULudine displays once again a very high level of performance compared to the other lubricants, even if they also can be considered as efficient. Indeed, with mean friction coefficients between 0.09 and 0.14 all along the test, without any detrimental variations which could be correlated to galling issues, those lubricants could be used in industrial conditions. Nevertheless, the performance level, if characterized by the only friction coefficient, is twice as high for the AFULudine product, providing for example additional process improvement capacities compared to usual lubricants.

Moreover, by obtaining equivalent ranking of the different lubricants (vs. LDR data), the present test results strengthen the representativeness of this tribometer with regard to the contact conditions occurring during stamping operations.

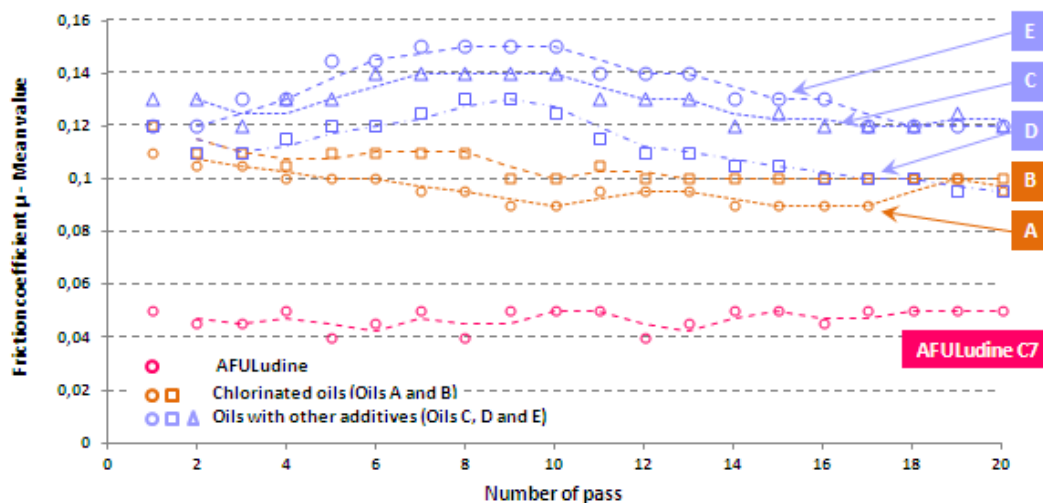


Figure 8. Evolution of mean friction coefficient versus number of pass (multi-pass strip drawing tribometer) – Comparison between different industrial oils with (orange – A and B) or without (blue – C, D and E) chlorine additive and with the AFULudine solution (C7 concentration -P1 process). 1.4509-K41 ferritic stainless steel grade. Doted lines correspond to sliding average (2 points) curves.

3.2. Industrial validation

3.2.1. Small batch production – Influence of the lubrication conditions

The figure 9 illustrates the evolution of the maximal force applied by the machine slide during stamping according to the number of parts produced. Whatever surface preparations (from P1 to P4), AFULudine solution induces more efficient lubrication conditions than the ones obtained with the current lubricant daily used by Cristel (Figure 9). Indeed, regardless of the lubrication method, the use of AFULudine is systematically associated with a significant decrease of the maximal force required to perform a part stamping, with a saving between 5,3% and 9,8% compared to the standard Cristel chlorinated oil, highlighting a high level of performance.

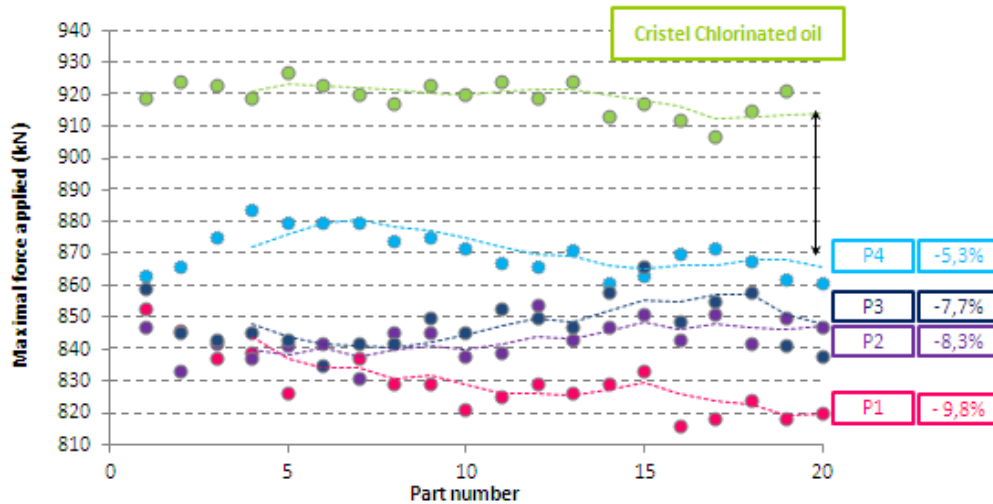


Figure 9. Evolution of the maximal force applied by the slide according to the number of pieces - Comparison between different AFULudine preparations, P1 (pink), P2 (purple), P3 (dark blue) and P4 (light blue), and the chlorinated oil used by Cristel (green). Dotted lines correspond to 4 points sliding average curves.

The differences observed between the P1 to P4 conditions are directly correlated to the quantity of alkylphosphonic acids molecules grafted on the metal surface and their organization, mainly depending on the associated process. As explained on the experimental section (2.3) the four preparation conditions differ, amongst others, by the contact time between the AFULudine solution and the metal surface, ranging from minutes to seconds. According to a previous study made on an equivalent 304 stainless steel [15], even if it may be reasonable to suppose that molecules immediately graft at the surface due to a spontaneous reaction, the contact time has been highlighted as having a significant influence on the layer quality. Indeed, Roy and al. [15] reported that increasing the grafting time induces an increase of the recovery rate, up to a critical point (for few hours of grafting time). It is not surprising therefore that performance variation occurs depending on the lubrication process used. Both concentration and contact time are so influencing the tribofilm efficiency.

Moreover, it is important to remind that, with a constant blank-holder pressure equal to 5.5MPa (Table 2), the production conditions are more severe than the ones used on the 20t press at the laboratory scale.

3.2.2. Series production – Influence of the temperature

While the previous small batch productions was not able to underline the inherent thermal effect of series production (increase of the temperature with the number of pieces), the bigger series (200 pans) carried out with the AFULudine solution (C1-P3) brought to light a transitional regime before reaching from the eightieth part a more stable working domain (Figure 10). During this transition period, different stop (from few seconds to few minutes) were carried out, mainly motivated by quality tracking. Each one of them is responsible for an immediate increase of the maximal force applied by the machine after restarting. The more symptomatic example is the force increase observed after the main stop done (5 minutes) at the piece 27. Without changing any process parameter, the only variable between the piece 27 and 28 is the tools temperature. A positive sensitivity of the AFULudine solution to the working temperature has been thus highlighted.

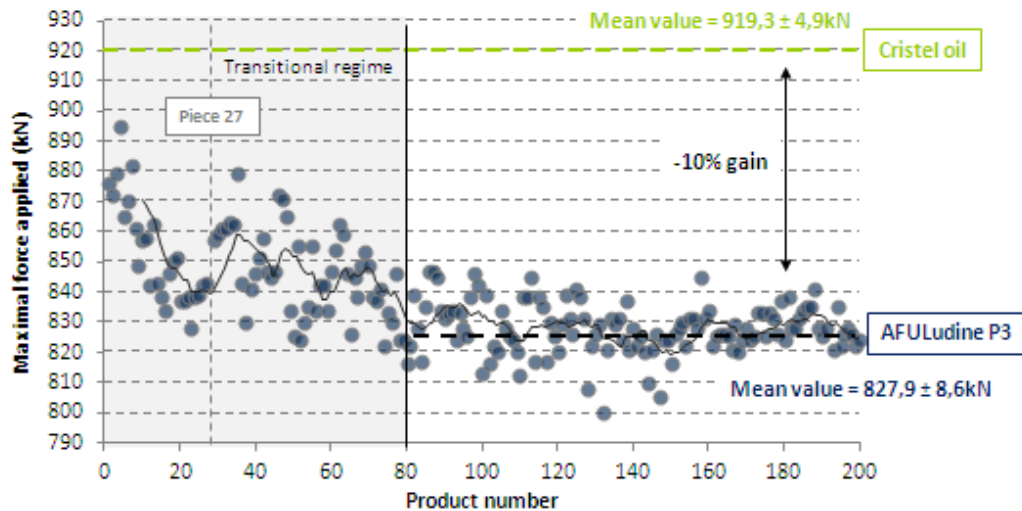


Figure 10. Evolution of the maximal force applied by the slide according to the number of product for a 200 pieces series production – AFULudine solution used with the C7 concentration and the P3 lubrication process. The dotted line corresponds to a 10 points sliding average curve.

More significantly, AFULudine use is leading once again to a significant gain in terms of working energy required to perform a piece: 10% compared to the current Cristel oil, with a mean maximal force applied by the slide equal to 827,9kN. Moreover, the 200 produced pans are perfectly acceptable in terms of quality, without generating any deterioration of the coated tools.

4. Conclusions

Several previous studies showed the effectiveness of alkylphosphonic acids to improve friction behaviour of different metallic substrates (Cu, Ti, Stainless steels...). Mainly representative of laboratory conditions (using different tribometers), the transition to an industrial scale, targeting more especially stamping operations, had not been investigated previously.

The present study goes further into the industrialization of the concept of the Self-Assembled-Monolayer through the AFULudine solution by providing results obtained on both laboratory and industrial presses. With a lower friction coefficient and/or force applied for carrying out stamped parts compared to other lubricant solutions, AFULudine appears to be relevant to offer an alternative to mineral oils and more especially to the chlorinated ones that highlight better performances than most non-chlorinated lubricants. Of course, AFULudine will not systematically replace these daily used lubricants which can be efficient or irreplaceable for specific technical or economical reasons. But for once, a more eco-friendly lubricant solution is conceivable. As illustrated by the collaboration with Cristel, AFULudine is well-adapted to mass production of stainless steel stamped parts even if technical parameters have to be well adjusted.

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