Influence of different external lubricants and their deposition mode on green nuclear fuel pellets during cold compaction

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In the framework of manufacturing future nuclear fuels, some solutions set out to optimize the nuclear powder compaction process. Lubrication is used in the fabrication process to reduce friction between the actinide oxide grains and press tools. A higher plutonium content and an isotopic composition with more ^{238}Pu for the purpose of plutonium multi-recycling would increase the level of radioactivity and the temperature in future UO_2 + PuO_2 powder mixtures. This temperature could be detrimental to maintaining the lubricant's properties within the mixture.
A solution to this problem could be to replace the internal lubricant by a lubricant deposit on the die wall (external lubrication) during compaction process. This lubrication technique, when combined with internal lubrication, is known to enhance the mechanical strength and density of the pellets produced by powder compaction.
This paper investigates the influence of this kind of lubricant deposition on the die wall for UO_2 powder pelletization without any admixed internal lubricant. Different lubricants and various ways of depositing lubricant on the die wall were investigated through different parameters during powder compaction and pellets characterization. We have thus compared depositing zinc stearate solid by lubricant powder pelletization with spraying. Results show that the tensile strength of green pellets is enhanced and surface defects are reduced when the lubricant is sprayed. Furthermore, the application of viscous oil on the die wall does not make it possible to manufacture UO_2 pellets, which is probably due to the tribological inefficiency of this form of lubricant under our conditions, while an industrial grease makes it possible to obtain green pellets with good properties and fewer surface defects. The lubrication mechanism changes from one lubricant to another and acts on the wall friction, the friction index, the ejection force, and the characteristics of the final green pellets. The ejection force seems to be more sensitive with respect to assessing the performance of a lubricant when only external lubrication is used in nuclear powder compaction.

Keywords: Lubrication, friction index, ejection force, wall friction, pellets, compaction, nuclear fuel, stearate, oil, die, uranium, density, grease, powder, defect, wear, tribology

1. Introduction

Internal lubrication of the powder is the widest lubrication method used in nuclear sector [1, 2, 3], as well as in fields of metallurgy powder [4, 5, 6], minerals [7] and pharmaceuticals [8, 9, 10]. This lubrication method enhances intergranular sliding and grain rearrangement, reduces inter-particle stresses, and extends the working life of costly tooling []. It is sometimes combined with the lubrication of the press die wall (external lubrication) in order to optimize the compaction process [, , 11]. The main advantage of external lubrication, when combined with internal lubrication, is that it reduces the amount of admixed lubricant added to the powder [, , 12] and enhances the compressibility of the powder at high pressure.
It is also known that the additional lubrication of the die wall during powder compaction increases the transmitted force during powder compaction and the green densities [,] while decreasing the ejection force of the pellets [, ,]. These advantages make it possible to improve the quality of the sintered pellet.
Different ways exist to deposit lubricant on the press die wall. Tribo-static charging of dry lubricant is the widest technique used to carry out this type of lubrication [, , , 13]. C. Machio *et al.* [14] recently used aqueous dispersion and lubricant paste to perform die wall lubrication for TiH_2 cold compaction. They observed an improvement in the mechanical properties and the quality of the sintered materials. In the literature, however, similar studies on nuclear fuel powders are not reported.
This study aims to investigate the influence of different forms of lubricant used during the cold compaction of UO_2 powder: zinc stearate in powder form and in spray form, silicon oil, and a specific industrial grease. This work was carried out by analyzing the friction indexes, wall friction forces, ejection forces, compressibility and aspect of the final green pellets. Besides comparing the different lubricants, this study also aims to identify a suitable parameter for selecting the best lubrication conditions. In these experiments, the nuclear powder was free of any admixed lubricant

during the compaction process.

2. Experimental set up

2.1. Raw material

A batch of dry processed UO_2 powder provided by ANF Lingen was used to carry out the experiments. It contained around 8.5% of U_3O_8 additives. The powder in Figure 1 shows a large particle size distribution (from 0.5 µm to 300 µm) with an overall super-stoichiometric oxygen content such that x = 0.13 in UO_{2+x}.

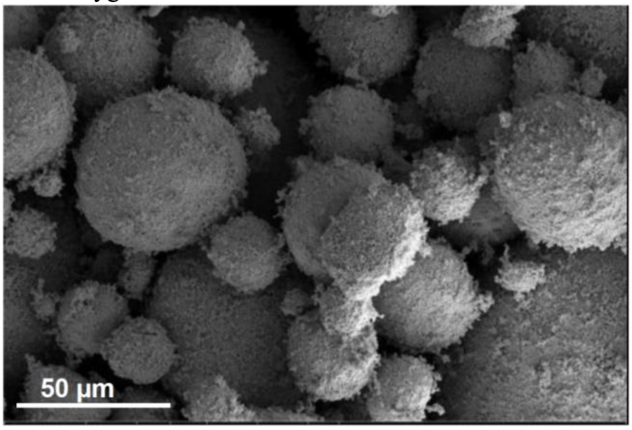

Figure 1: UO_2 powder particles observed by scanning electron microscopy (SEM)

2.2. Lubricants

The experiments were carried out with four different commercial lubricants: zinc stearate powder, zinc stearate spray, silicon oil, and an industrial grease.

2.2.1 Zinc stearate powder

Zinc stearate (StZn) is widely used as lubricant in the powder compaction of nuclear fuels, as well as being used for other applications [, , , 15, 16]. It is a metallic salt of fatty acid formed by two long aliphatic carbon chains with a metallic atom in the center. Its melting point is around 120°C. The powder is composed of agglomerates between 20 and 40 µm in size, which form layers of StZn less than 5 µm in size (Figure 2). The lubricant batch used in our study was provided by Alfa AESAR.

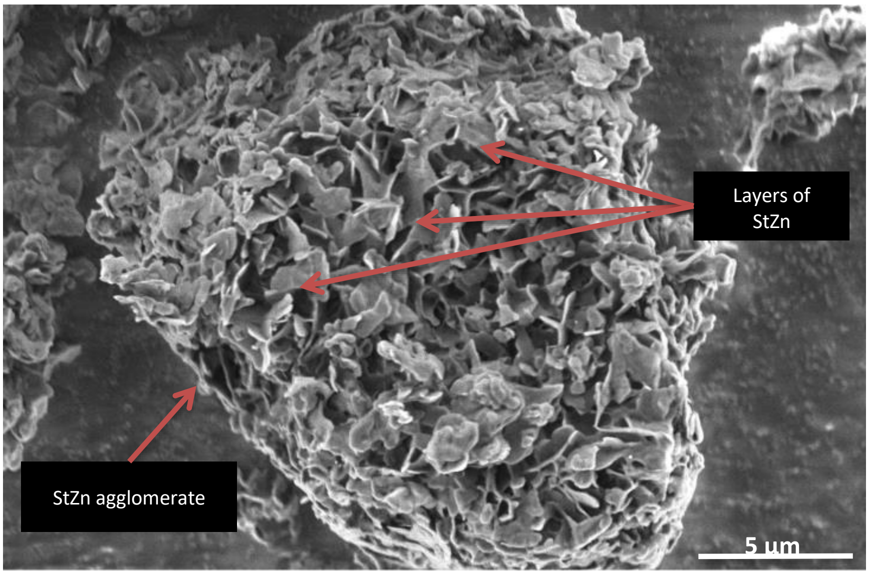

Figure 2: Example of an agglomerate of zinc stearate constituted by particles of StZn in form of layers observed by SEM technology.

2.2.2 Zinc stearate spray

This universal release agent is often used as a lubricant during the forming process in different industrial fields, as well as being used for decorative purposes. It is used to deposit solid coating on surfaces in order to protect them against wear and friction phenomena.

The commercial zinc stearate spray used in this work contained some volatile organic elements such as: 1,1-difluoroethane, dimethyl ether and dichlorofluoroethane, which evaporated around two minutes after lubricant deposition. The amount of these elements was not specified. However, it can be seen that the coating formed by the StZn spray takes on a specific aspect because of these elements (Figure 3), with the presence of a quasi-continuous film which is not observed in the case of StZn powder deposit. The commercial StZn aerosol used in this study was manufactured by IMS.

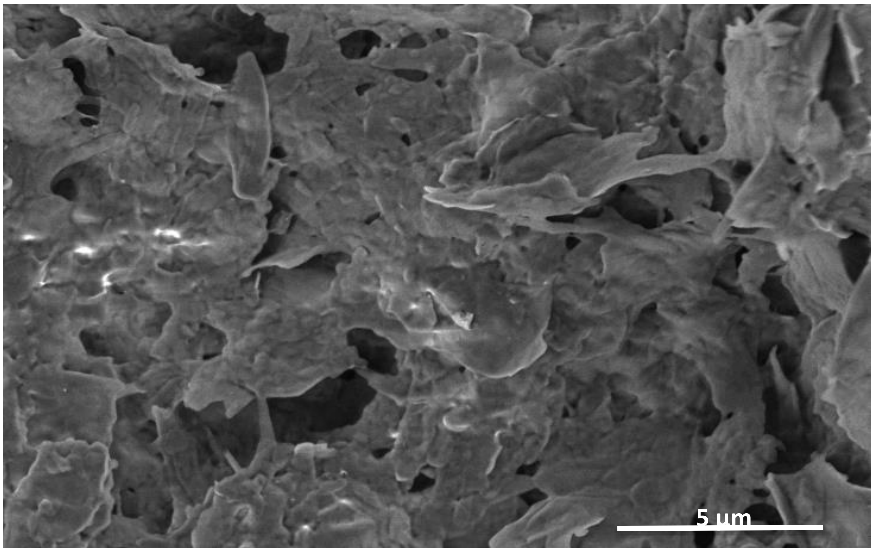

Figure 3: Structure of zinc stearate spray deposited on a surface and observed by SEM

2.2.3 Silicon oil

Silicon oil is used as a lubricant or release agent in several industrial fields due to its numerous qualities: it is chemically inert, reduces corrosion, shows little variation in viscosity with the temperature, etc. The silicon oil used in this study was a Rhodorsil® 47 V 1000 Oil with a viscosity of 1000 mm^2/s. This oil contained less than 0.5% of volatile agents.

2.2.4 Industrial grease

We used a versatile lithium-saponified grease based on a mineral oil. It showed good resistance to oxidation and good behavior at lower temperatures. The viscosity of the oil base at 40°C was around 100 mm^2/s. However, the viscosity of the grease as a whole was higher than 1000 mm^2/s at 25°C.

2.3. Compaction and lubrication methods

2.3.1 Lubrication methods

The StZn powder was deposited using the technique illustrated in Figure 4. It consists in manufacturing compacts with StZn powder. After ejection from the mold, a layer formed by the powder of lubricant particles covers the die wall.

An StZn aerosol was sprayed manually on the die wall by pressing the pump three times with a similar finger pressure. The silicone oil and the industrial grease were deposited using a dry cotton swab.

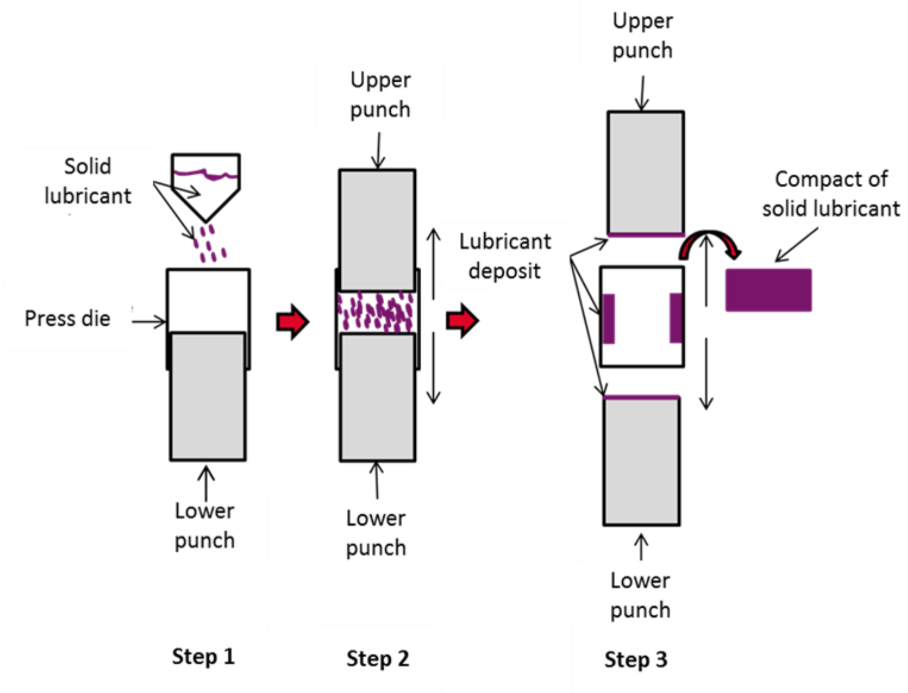

Figure 4: Deposition technique for StZn powder on the die wall

2.3.2 Compression method and data

The instrumented press used in this study came from INSTRON. The mold diameter was 10 mm and the roughness (Ra) of the die's inner tungsten carbide surface was 0.4. The lower punch and the mold were fixed during the compaction process, while the upper punch was brought into contact with the powder in order to compress it. The height of the chamber was fixed at 3.5 mm. The displacement speed of the upper punch was set at 2.1 mm/s. These compaction parameters allowed us to obtain a green compact with a H/D ratio close to 8/10, where H is the height and D is the diameter of the compact.

The friction index was calculated by Equation 1, including Janssen's constant K and the friction coefficient n_w []. This parameter takes into account the geometry of the pellets, and the applied and the transmitted forces which were obtained thanks to load cells located on the punches. The friction index takes into account transgranular friction and friction between the pellet surface and the die wall surface. A large friction index is known to be detrimental to the homogeneity of the compact characteristics due to unsuitable lubrication [].

$$\textit{Friction index} = n_w K = \frac{S}{ph} \ln\left(\frac{\sigma a}{\sigma t}\right)$$

Equation 1

where S is the surface of the compact, p is the perimeter, h is the height, σ_a and σ_t are the stress applied by the upper punch and the stress transmitted to the lower punch during compaction respectively.

The transmitted force ratio, σ_t/σ_a, is also a parameter that can be used to explain the phenomena occurring at the powder/die wall interface. In accordance with the literature, a high transmitted force is attributed to an increased plastic flow and radial movement of particles during compaction [].

The wall friction force, σ_w, corresponds to the difference between the force applied to the upper punch and the force transferred to the lower punch. This parameter takes into account friction phenomena occurring at the interface between the compacted powder and the die wall. In this study, it was calculated by an indirect method given by Equation 2 [17, 18, 19]. As in the case of the friction index, high wall friction can be associated with insufficient lubrication conditions, which induces high stress gradients within the pellets. Different methods can reduce the wall friction force [], like applying a lubricant on the die wall.

$$\textit{Wall friction force} = \sigma_w = \sigma_a - \sigma_t$$

Equation 2

The ejection force of the pellets after powder compaction is also widely used to assess the efficiency of the die wall lubrication process [20]. This parameter is known in powder technology field to be the main factor responsible for the final state of the compacts after demolding [,]. This force is directly recorded by the sensor placed under the lower punch.

2.3.3 Density measurement and green pellet surface characterization

The density was calculated on the basis of a cylindrical geometry by dividing the mass of the compact by its volume during compression and after demolding.

The surface of the pellets was mainly characterized by SEM observations and analyses.

2.3.4 Mechanical properties

Indirect tensile tests were conducted on the green compacts by means of Brazilian tests [21, 22] (Figure 5).

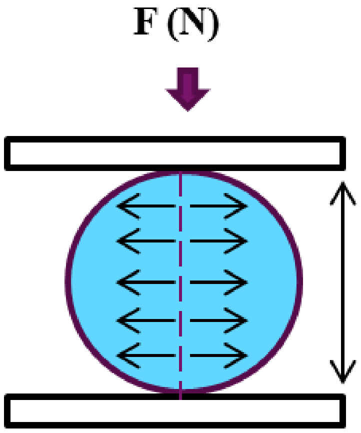

Figure 5: Schematic diagram of the Brazilian test principle

An indirect tensile test involves applying compressive loads to a cylindrical specimen, which act parallel along the vertical diametrical plane as shown in Figure 5. To distribute the load and maintain a constant loading area, the compressive load is applied through a wide stainless steel punch. This loading configuration develops a relatively uniform tensile stress perpendicular to the direction of the applied load and along the vertical diametrical plane, which ultimately causes the specimen to fail by splitting or rupturing along the vertical diameter (Figure 5) [23]. The indirect tensile strength of a cylindrical sample with a diameter D and thickness t is given by Equation 3.

$$\sigma_t = \frac{2F}{\pi D t}$$

Equation 3

where F is the failure load, and t the thickness or the sample length.

Twenty specimens of UO_2 compacts were taken of each lubricant to produce a suitable statistical analysis. Data analyses using Weibull's statistical law [24] were used to obtain the failure probability, Pr, of the compacts (Equation 4).

$$P_r = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right]$$

Equation 4

where m is the Weibull modulus which provides the width of the distribution, σ the distribution of material strength under tension, and σ_0 the characteristic Weibull strength.

3. Results and discussions

3.1. Characterization during compression

3.1.1 Transmitted force ratio

The ratio of the transmitted force to the applied force for the different lubricants evolved differently during compression (Figure 6).

StZn spray vs. StZn powder

From a normal load of 0 to 10 kN, the transmitted force ratio increased rapidly until reaching a given value for both lubricants This variation at lower pressure has already been observed for the compression of coal logs, with lubricated die wall [], alumina, and agglomerated ceramic powders [25].
For the zinc stearate spray, the transmitted force ratio remained constant at around 0.73 to the end of the compression process with a load of 10 kN. This was not the case of the zinc stearate powder where the transmitted force ratio decreased progressively from a ratio of 0.8 to around 0.69.
The transmitted ratio is therefore higher for the StZn powder in the case of a lower applied force. In this range of the applied forces, this means that radial stress transmission to the die wall is higher for the StZn powder; the presence of StZn grains better reduces the friction between UO_2 powder and the die wall than the coating formed by the StZn spray. This may be explained by the presence of a greater number of StZn particles for the powder lubricant at lower pressures.
The transmitted force ratio is higher for the StZn spray at higher applied forces, as the corresponding curve can be seen to remain constant while the curve for the StZn powder decreases progressively. Radial stress transmission to the die wall is reduced due to the presence of the StZn spray coating which resists the high pressure conditions, and therefore the force transmission from the upper punch to the lower punch is enhanced. On the contrary, the StZn powder can be separated from the die wall due to the significant size of its agglomerates.

Industrial grease vs. silicon oil

The variations in the transmitted force ratio for the industrial grease and for the silicon oil are similar to those of the zinc stearate spray. The stress transmitted is higher in the case of the industrial grease during the whole compression process. This higher stress transmission is attributed to better frictional dissipation at the die wall [,]. In this case, the shearing stress at the UO_2 powder/die wall interface uses less energy, and therefore the force transmission from the upper punch to the lower punch is enhanced.

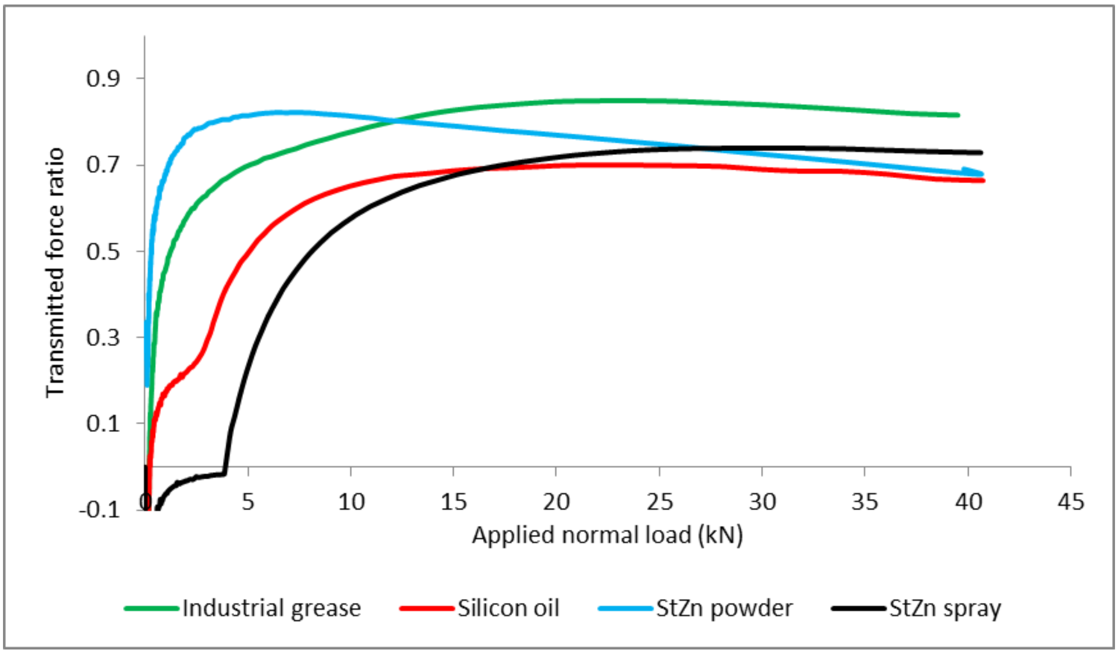

Figure 6: Transmitted force ratio for UO_2 compaction with different external lubricants

3.1.2 Wall friction force σ_w
The wall friction force calculated from the applied and transmitted forces during the compression of UO_2 powder (loading and unloading steps) for all the lubricants is shown in Figure 7. In this work, we assumed that the absolute wall friction represented the friction phenomena.
As expected, the absolute wall friction force increased almost linearly with the normal force for all the lubricants during the loading step. This behavior of the wall friction force was also observed with the compression of coal logs [] and bentonite powder [].
For each lubricant, the absolute friction force decreased almost linearly with the normal force down to zero during the

unloading step. This absolute value then increased due to the compact rebound due to the increased contact surface between the compact and the die wall. At a certain value, the absolute friction force decreased once again because the increase in the contact surface slowed down.
The hysteresis observed between the loading and unloading forces is attributed to a reduction in the radial stress due to elastic rebound in the axial direction and due to contraction in the radial direction during unloading.

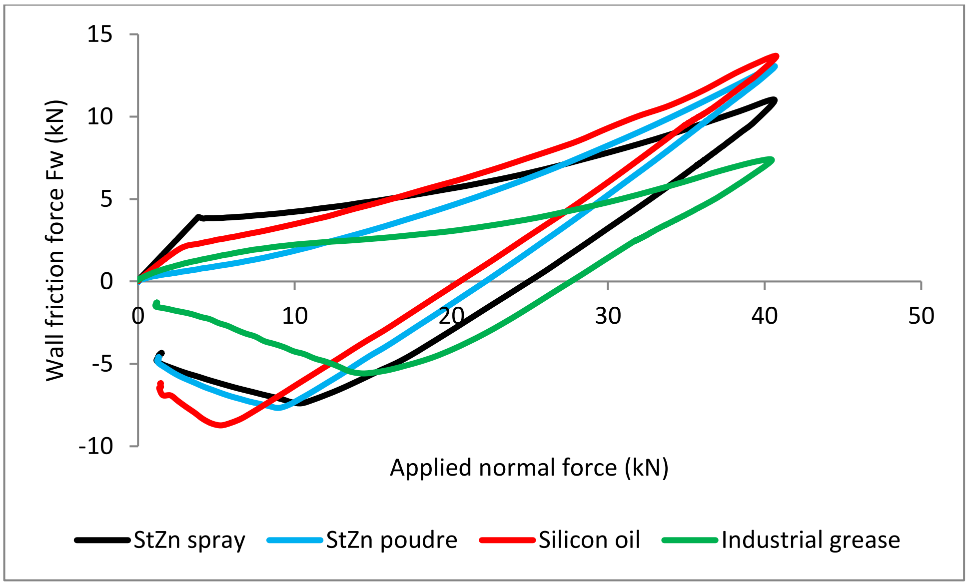

Figure 7: Wall friction force during loading and unloading for compaction of UO_2 powder with different external lubricants

3.1.3 Friction index
Figure 8a shows the variation in the friction index, n_wK, plotted against the applied compaction stress during the compaction process for the four external lubricants.

StZn spray vs. StZn powder

It can be seen that the curve for the StZn spray remains stable during the compression process, contrary to that of StZn powder; this difference may be attributed to their lubricity.
It can be seen that n_wK (StZn powder) < n_wK (StZn spray) from 0 to 150 MPa, and that n_wK (StZn powder) > n_wK (StZn spray) above 150 MPa. The transmitted force ratio decreases from 150MPa due to an increase in the wall friction forces in the case of the StZn powder as remarked previously. As a consequence, the friction index increases as shown in Figure 8. This means that lubrication is limited in this case and that friction phenomena on the UO_2 powder/die wall interface decrease the transmission of the force. The StZn powder particles in the interface do not withstand the high pressure well, and the friction index increases as explained for the transmitted force.
Conversely, the wall friction forces and the transmitted force ratio remain stable for the StZn spray due to the better and more effective lubrication. The coating formed by the StZn spray resists higher pressure and the friction index is therefore less affected. It can be said that effective lubrication provides sufficient densification and the friction index remains constant [].

Industrial grease vs. silicon oil

The difference between the friction indexes of grease and silicon oil was due to the difference in viscosity between the two lubricants; during the whole compression process, n_wK (silicon oil) > n_wK (Industrial grease). The viscosity of the lubricant is known to improve the rheological and tribological properties of oil-based lubricants and to reduce friction phenomena, particularly in mixed lubrication [26, 27]. A lubricant with a higher viscosity leads to reduced friction. In the case of the industrial grease, contact between the UO_2 powder and the die wall was reduced and the process required less energy to shear the material. Consequently, the wall friction force was lower and the friction index decreased.

Viscous lubricant vs. zinc-stearate-based lubricant

In the cases of silicon oil and industrial greases, the variation in the friction index differs from that of zinc-stearate-based lubricants. The friction index decreases progressively and reaches a steady state at a compaction stress of 150 MPa for viscous lubricants. This result is explained by the fact that UO_2 powder densification develops at a slower rate when industrial grease and especially silicon oil are used compared with zinc-stearate-based lubricants []. There may also be other possible explanations for the behavior of the silicon oil and the industrial grease at lower pressure, compared with zinc-stearate-based lubricants. It is true that while the mold is being filled, the first particles of UO_2 powder near the die wall drive the lubricant to the bottom of the die and create contact between the rest of UO_2 powder particles and the die wall in a boundary lubrication regime. This situation may increase friction phenomena at the UO_2 powder/die wall interface with lower pressure. Compared with zinc-stearate-based lubricants, UO_2 particles may mix with the lubricant at the interface due to their viscous nature and hinder grain displacement to cause additional friction when the compaction pressure is lower.

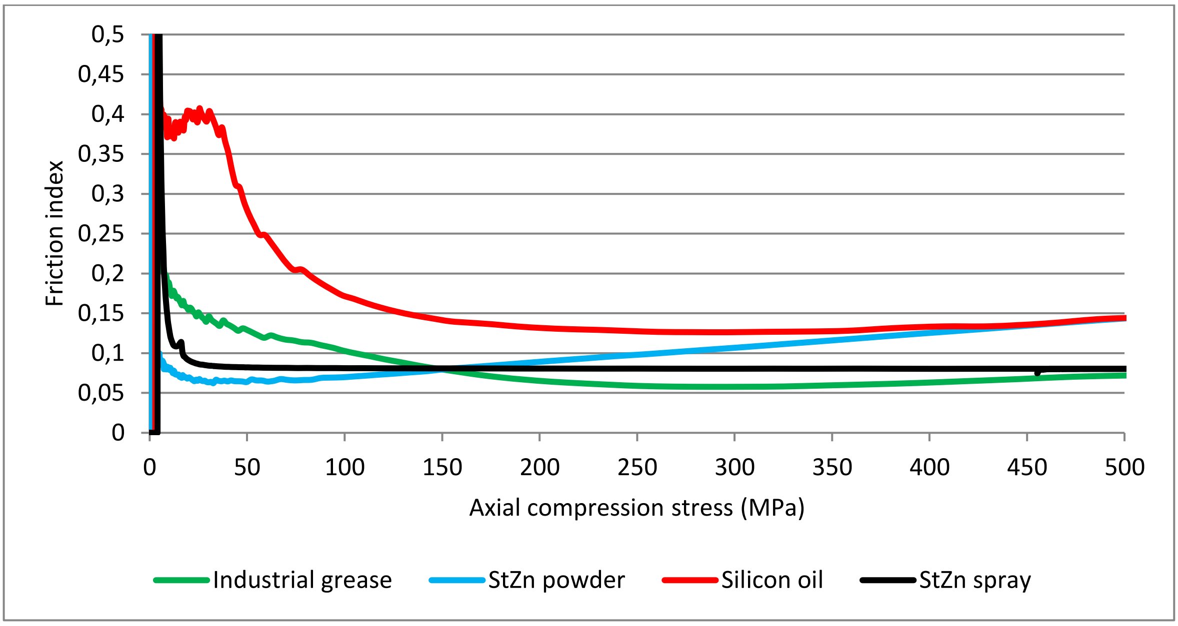

Figure 8: Friction index plotted against the compaction stress

3.1.4 Ejection force

The ejection forces of UO_2 pellets recorded for the different lubricants are shown in Figure 9. The difference between the ejection forces corresponding to the different lubricants is important and monotonous from the beginning to the end of the ejection force.

The ejection force is around 55% lower in the case of the StZn spray used as a lubricant, compared with the experiment using StZn powder. This result may be due to the fact that the coating formed by the StZn spray on the die wall generates a mixed lubrication regime during the demolding of the UO_2 pellets, with contribution from the organic products, while the tribofilm formed in the case of the StZn powder does not create the same lubrication regime as that for the StZn spray. This probably explains why the manufacturing residues and the 'squealing' noises due to friction between the formed pellet and the die wall, are less significant or non-existent when the StZn spray is pulverized on the die wall.

The ejection force curve for the silicon oil could not be provided due to the strong 'squealing' noises that occurred when the UO_2 pellets broke during ejection. Thus, it can be said that this lubricant does not form a tribofilm protecting the die wall during ejection. It confirms the fact that the silicon oil may have been removed during compression, which leads to direct contact between the UO_2 powder particles and the die wall.

The industrial grease obtained the lowest ejection force. Compared with the silicon oil, the squealing noises were much smaller during ejection in this case. This difference in tribological behavior may be due to the difference in viscosity between the silicon oil (lower) and the industrial grease (higher) []. First of all, the lubricant properties seem to be linked to the viscosity, as explained in the previous section. Furthermore, lubricant mobility can be affected by viscosity which affects the contact between the powder and the die wall. This phenomenon is enhanced during the ejection stage which occurs after compression (i.e after a stage which can cause the lubricant to shift).

Furthermore, the ejection forces differ greatly from one lubricant to another. This parameter seems to be relevant for assessing the different lubricants and deposition modes in nuclear pellet compaction. It provides a good assessment of the performance of the different lubricants in nuclear fuel compaction.

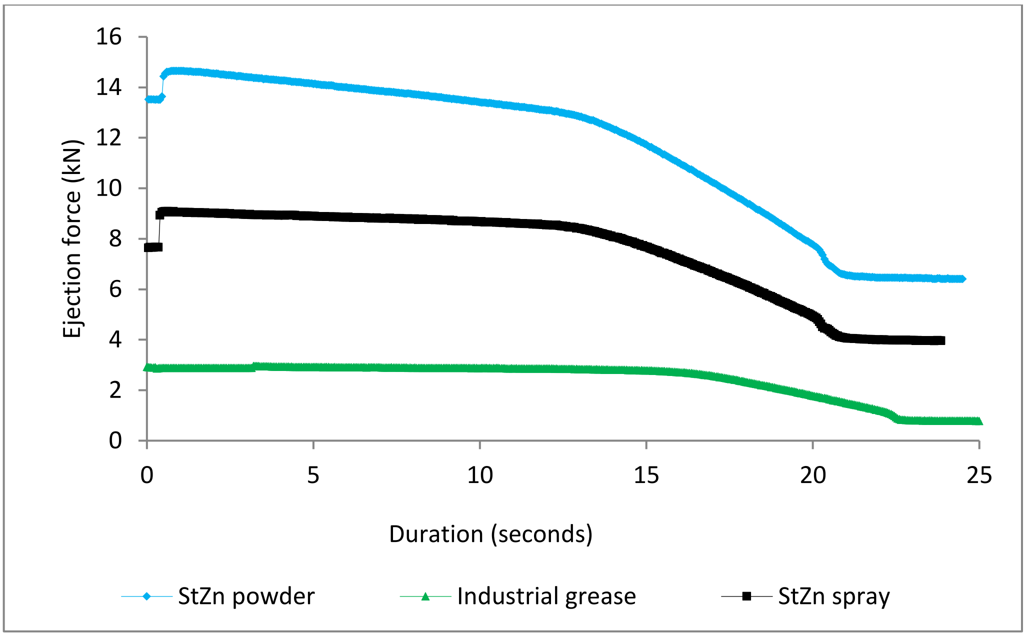

Figure 9: Variation in the ejection force of the green compacts for different lubricants

3.1.5 Compressibility

Figure 10 shows the variations in the density of the powder during the compaction process for each lubricant. The curves highlight no significant difference in the UO_2 powder compressibility between each lubricant tested. This result is in good agreement with studies reported in the literature on other materials like metallic powder [,]. Nevertheless, it has been confirmed that UO_2 powder densification is a bit lower in the case of silicon oil, probably due to the insufficient lubrication conditions.

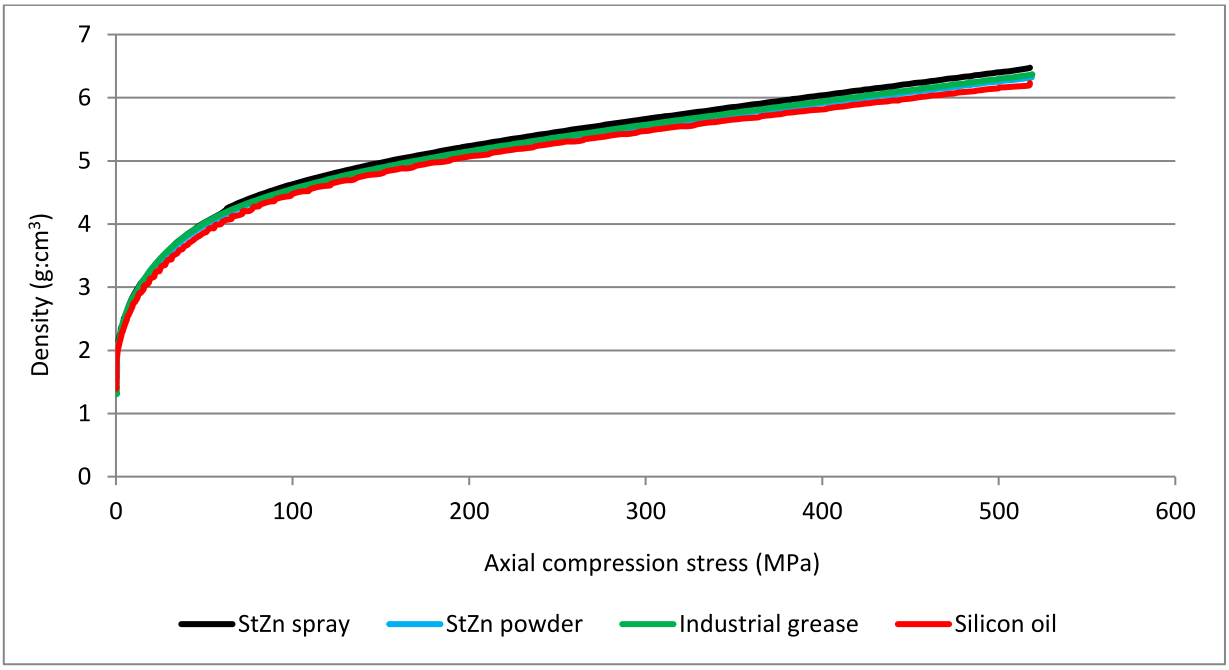

Figure 10: Compressibility of the UO_2 powder during the compression process for the different lubricants

3.2. Green pellets characterization

3.2.1 Compactibility

Table 1 shows the mean value of the green pellet densities after ejection (20 pellets for each lubricant). As in the case of compressibility, the form or type of lubricant have very little influence on the compactibility of the green pellets. In the case of silicon oil, however, entire compacts could not be obtained because of stress accumulation during demolding. Thus, this lubricant seems to be prohibitive for UO_2 powder compression.

Table 1: Densities of green pellets after ejection from the mold

Lubricants	Density of green pellets (g/cm^3)
StZn powder	6.1 ± 0.06
StZn spray	6.2 ± 0.11
Industrial grease	6.2 ± 0.04
Silicon oil	Broken

3.2.2 Surface characterization

In order to better understand the behavior of the different external lubricants on the green pellets, SEM images of pellet surfaces were taken as shown in Figure 11. These observations were not possible in the case of silicon oil where the pellets were completely broken. In good agreement with the results of the ejection forces, residual parts of the tribofilm formed by the StZn spray are visible on the surface of the pellets after ejection, contrary to the StZn powder which is much less present. In the latter case, some surface defects can also be seen on the pellet: micro-ploughing phenomena corresponding to the loss of some UO_2 grains or particles of StZn powder which occupied voids during compaction and micro-cracks. This is not the case when the StZn spray is used.

It is worth pointing out that a grease-soaked area can be observed macroscopically almost 2 mm from the surface through to the bulk of the pellets when the industrial grease is used as the lubricant. This grease-soaking phenomenon forms a thin layer, corresponding to a mixture of powder and grease on the surface of the pellets, which is subject to scuffing and tearing after ejection, as observed in Figure 12. Except for the scuffing phenomenon occurring at the surface, there are no other important types of defects. The thin layer formed probably reduces friction between the pellets and the die wall during ejection.

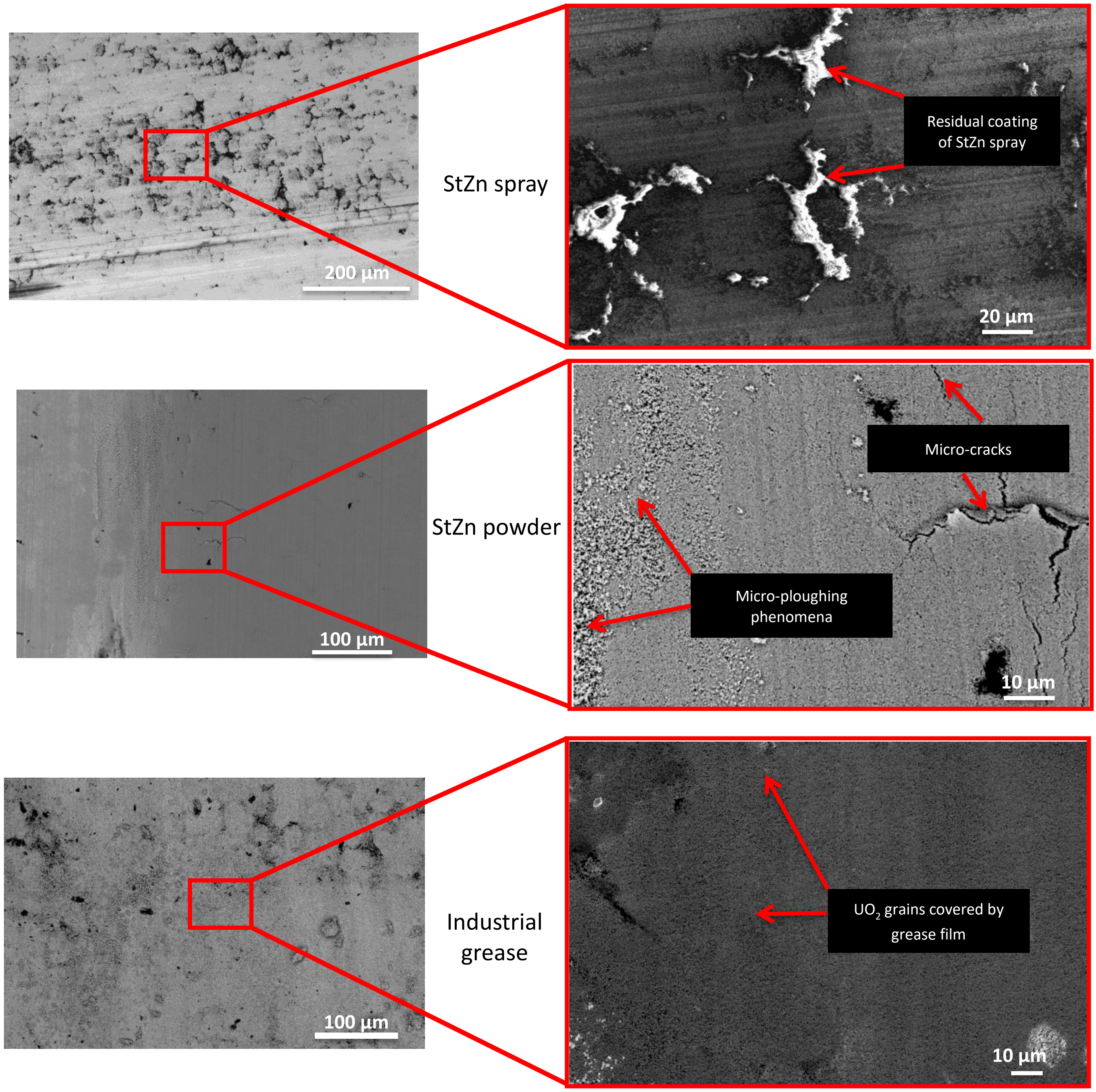

Figure 11: SEM images of pellets surfaces for the different forms of lubricants

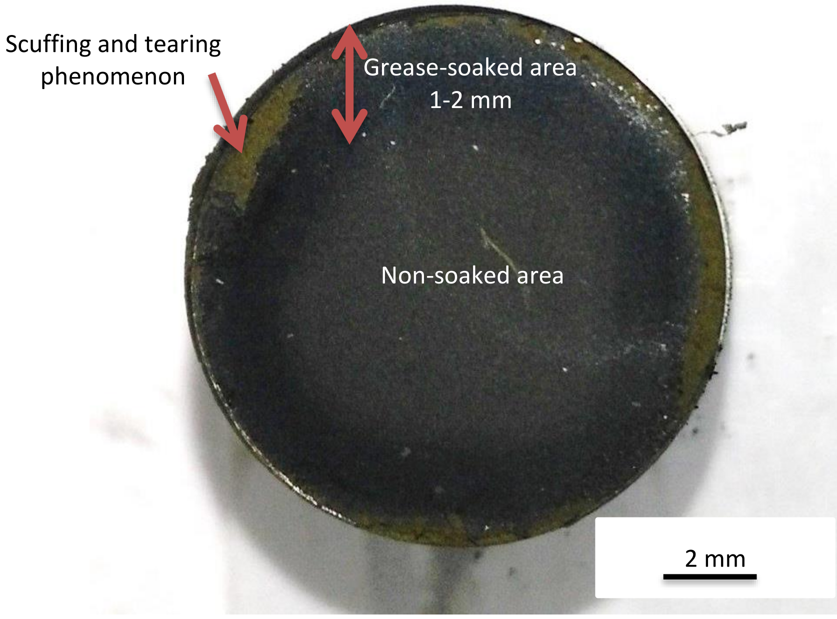

Figure 12: Top view of a UO_2 pellet obtained in the case of external lubrication with industrial grease

3.2.3 Mechanical properties

The tensile yield stress (σ_0) and the Weibull distribution factor *m* of the compacts for the different lubricants are shown in Table 2. These results are in good agreement with the aspect of the UO_2 pellets and particularly with the ejection forces; the green strength is higher for the industrial grease and for the StZn spray, compared with the StZn powder. Nevertheless, the green pellet tensile strength for the StZn spray and the industrial grease are quite similar. This kind of similarity is also observed in the literature for metallic powder compression using an external lubrication technology []. The results are typical of brittle materials because of their low density.

Table 2: Mechanical properties and densities of UO_2 green compacts for the different forms of lubricant

Lubricants	**Dispersion factor m**	**Tensile yield strength σ_0 MPa**
StZn powder	8.2	1.2
StZn spray	8.7	1.8
Industrial grease	4.8	1.7
Silicon oil	----------------	Broken

4. Conclusion

External lubrication of the die wall in the UO_2 powder compaction process is beneficial for the resulting green compacts. This study shows that how the lubricant is applied may have some consequences on the quality of the UO_2 green compacts. More specifically, lubrication with a StZn spray generates less friction with better ejection and few surface defects, contrary to lubrication with a zinc stearate powder. Better lubrication conditions are obtained when the layer formed by the sprayed lubricant remains on the die wall during demolding and reduces the ejection force by facilitating sliding between the compact and the die wall. The application of silicon oil is prohibitive for the compression of UO_2 powder and causes green pellets to break. Furthermore, industrial grease makes it possible to obtain UO_2 green pellets with very few surface defects and with interesting properties. This study shows that the friction index and the wall friction ratio provides some information on the lubricants, but the ejection force seems to be the most sensitive parameter that needs to be taken into account when assessing different external lubricants for UO_2 powder compaction.

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