

# **EFFECT OF COMPACTION TEMPERATURE ON THE LUBRICANT DISTRIBUTION IN POWDER METAL PARTS**

**M. Gagné, Y. Thomas\* and L.P. Lefebvre\***

**Rio Tinto Iron and Titanium - Technology  
Tracy, Québec, Canada**

**\*Industrial Materials Institute /National Research Council Canada  
Boucherville, Québec, Canada.**

## **ABSTRACT**

Warm compaction is a process leading to parts with high green strength and density. An increased compacting temperature lowers the flow stress of the metallic particles and increases their ductility, resulting in higher powder compressibility and stronger interparticles bonding, either mechanical or metallurgical. Although many studies have been published on warm compaction, very few investigated the influence of compaction temperature on the behavior of the lubricants. In most cases, the choice of a lubricant for warm compaction relies only on the experimental evaluation of the compressibility and of the ejection performance of the selected metal/lubricant system.

An investigation was carried out to characterize the behavior of the lubricant during the compaction of water-atomized iron powder mixes at 25 and 150°C. Using techniques such as SEM, WDS and SIMS, the microstructure of the compacts and the distribution of the lubricant within the parts were characterized for mixes containing different lubricants (LiSt and EBS). The results were related to the properties of the iron powders and lubricant during the compaction of the parts.

## **INTRODUCTION**

The density level that can be achieved when compacting metallic powders under a given pressure is a function of the compactibility of the powder, of the friction between the powder particles and the die walls and of the springback of the compacted part after ejection. The compacting behavior of a powder mix is an intrinsic characteristic which depends on composition, particle shape and size distribution while springback is mainly dependent on the elastic properties of the pressed materials. On the other hand, to achieve a high density, the friction forces at the die walls need to be minimized during the compaction as well as during the ejection of the parts. A lubricant is generally admixed to the metallic powder to impede the formation of metallic contacts at the die/particles interface during compaction and

ejection. However, the amount of lubricant needs to be minimized in order to reach the high density level required to maximize the green and sintered properties of the parts.

The warm compaction process enables the fabrication of parts with high density and green strength. It is recognized that increasing the compacting temperature lowers the flow stress of the metallic particles and increases their ductility, resulting in higher powder compactibility [1,2]. The properties of the lubricants being temperature sensitive, the temperature rise may also affect the behavior of the lubricants and the compaction and ejection processes [3,4]. It has been suggested in the literature [5] that the increased fluidity of the lubricant at higher temperature results in a stronger tendency to flow from the pores towards the outside of the part, enhancing the ability of the powder itself to be densified and improving the lubrication at the interface between the powder and the die walls.

A study was conducted to identify the effect of the compacting temperature on the densification and the lubricant behavior during the manufacture of green parts. The porosity and lubricant distributions in green iron powder compacts were evaluated after compaction at 25°C and 150°C ; two lubricants with very different physico-chemical and rheological properties were tested. The lubricant distribution was analyzed using different techniques (SEM, WDS, SIMS) and the results were interpreted with respect to the porosity distribution in the compacts.

## **EXPERIMENTAL PROCEDURE**

### **Sample Preparation**

A high purity water-atomized iron powder<sup>1</sup> with a particle size smaller than 250 μ was used in this study. The powder was dry-mixed with 0.75 wt% lubricant in a V-type blender for 30 minutes. Mixes containing lithium stearate<sup>2</sup> (LiSt) and ethylene bisstearamide wax<sup>3</sup> (EBS) were prepared. LiSt and EBS lubricants show a solid-liquid transition at about 230°C and 145°C, respectively.

In this study, the test specimens which consisted in standard transverse rupture bars (T.R. bars) (3.175 x 1.27 x 0.635 cm<sup>3</sup>) were pressed in a double action tool steel die using an hydraulic press. As seen in Table I, most specimens were compacted at 620 MPa either at room temperature or 150°C. For comparison purpose, specimens of pure iron powder were fabricated using die wall lubrication (graphite spray) at both temperatures. Samples with a targeted density of 7.20 g/cm<sup>3</sup> (density obtained with the material containing LiSt compacted at 25°C/620 MPa) were also prepared via compaction at 150°C to evaluate the effect of the density on the distribution of porosity and lubricant. Densities were measured on three T.R. bars from each set of experimental parameters using the weight (± 0.0001 g) and the physical dimensions (± 2.5 μ) of the bars.

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<sup>1</sup> *ATOMET 1001HP* supplied by Quebec Metal Powders Ltd.

<sup>2</sup> *Lithium stearate 200* from H.L. Blachford Ltd.

<sup>3</sup> *Atomized Acrawax C* from Lonza Inc.

## Microstructural Characterization

Density and lubricant distributions were characterized on specially prepared specimens using different analytical techniques. For those analyses, the specimens were sectioned and mounted in an epoxy resin. In order to limit the possible interactions between the lubricant and the polishing media, the mounted samples were first dry ground manually up to the 600 grit abrasive paper and were then manually polished with an oil-suspension of diamond. To remove the water-soluble oil from the polished surface, the specimens were gently rinsed with water. The efficiency of the rinsing procedure to remove the oil from the surface of the specimens and from the pores was confirmed with SIMS analysis.

The distribution of porosity of the as-compacted structure was characterized qualitatively with a JEOL JSM 5800 scanning electron microscope. This was done by examining the polished cross-sections of the specimens at the center and on the edge of the bars.

High resolution microprobe analyses were performed with a Cameka SX500 microprobe equipped with a high-resolution X-ray wavelength detector to determine the lubricant distribution in the samples. Two different techniques were used to evaluate the lubricant distribution. First,  $C_{K\alpha}$  maps (300X) were taken at the center and on the edge of the different samples ( $V = 10$  kV and  $I = 45$   $\mu$ A,  $t = 8$  minutes) and were compared. In the second series of analyses, sixty-four C measurements (area of analysis =  $80$   $\mu$  x  $100$   $\mu$ ) were taken along the central axis of the specimens over a distance of  $\sim 6$  mm., i.e. from the center to the edge of the cross-section of the samples. For those analyses,  $C_{K\alpha}$  radiations were counted for 8 minutes ( $V = 10$  kV,  $I = 55$   $\mu$ A).

Secondary Ion Mass Spectroscopy analyses were also performed to evaluate the lubricant distribution in the samples. In this study, the  $^7\text{Li}$  ions were used to map the LiSt distribution on the sample cross-section. A Cameka IMS 4f magnetic sector dynamic SIMS with a field of view of  $150$   $\mu$  or  $250$   $\mu$  diameter was used.  $\text{O}_2$  primary ion beam with a primary beam current of 800-1000 nA was utilized for an acquisition time of 45 s. Five images were acquired from the edge and central regions of each sample. They were converted to a binary image using a threshold operation. The threshold used was that determined by the computer for all images. Although some interparticle information was lost, it did not significantly affect the results. The total area occupied by lithium was determined from each mapping. The mean and standard deviation values were calculated for each area and sample.

The distribution of the lubricant on the surface of the samples was also evaluated after ejection. Top and sliding surfaces were examined with a JEOL JMS 6100 scanning electron microscope. Weight loss during delubrication was measured to evaluate the amount of admixed lubricant remaining in the part after compaction. For that purpose, one specimen from each experimental condition was slightly polished to eliminate the lubricant on the sample surface and then sintered at  $1120^\circ\text{C}$  during 30 minutes in dissociated ammonia to remove the lubricant from the compacts. Weights before and after sintering were measured.

## **RESULTS AND DISCUSSION**

### **Density Measurements**

The effect of the compaction parameters (pressure and temperature) and mix composition on the green density of the bars is presented in Table I. Higher density was achieved when the powder was compacted with die wall lubrication either at room temperature or at 150°C. Admixing the powder with a solid lubricant reduced the green density by 0.06 to 0.08 g/cm<sup>3</sup>.

The mechanisms that may explain the lower density achieved when admixing a lubricant to the powder are :

- Higher friction between the compacts and the die walls may occur with admixed lubricant compared to graphite die wall lubrication. This effect does not appear significant at room temperature since similar densities are achieved in iron/LiSt samples compacted with or without die wall lubrication (C and C'). At high temperature, previous work [4] has however shown that the lower ability of some metallic stearates to reduce the friction at the die walls may affect the ultimate density of the compact.
- The presence of internal lubricant may lead to larger springback after ejection, due either to the minimized interparticle cold welding or to the springback of the lubricant itself. As seen in Table I, for samples made at 25°C, the length of the compacts fabricated with admixed lubricant is slightly larger than that of the specimens made without admixed lubricant. This suggests that the springback is smaller when no admixed lubricant is used. However, the effect of LiSt on the springback is less important at 150°C and the variation in springback cannot explain the large difference in density observed.
- The lubricant may impede pore closure during compaction. The admixed lubricant is easily deformed; it thus flows between the iron particles and fills the pores present in the sample during compaction. The presence of lubricant in the compact may then impede the closure of the pores during compaction. It is believed that this phenomenon is mostly responsible for the difference in density measured between the specimens with and without lubricant.

When the compaction temperature increases from room temperature to 150°C, the density of the samples pressed with die wall lubrication increases from 7.25 g/cm<sup>3</sup> to 7.38 g/cm<sup>3</sup>. It is also seen that compacting the steel/LiSt mix at 150°C and 480 MPa is equivalent to compacting this blend at room temperature and 620 MPa. The increased compressibility can be related to the enhanced plastic deformability of the metallic powder as the temperature increases. As previously stated [2] increasing the compacting temperature lowers the flow stress of the particles and allows a high level of deformation and mutual accommodation between the particles.

Although the rheological properties of LiSt and EBS are significantly different at 150°C (EBS is liquid while LiSt is solid at that temperature), the compaction at 620 MPa/150°C of mixes containing those lubricants leads to parts with similar densities. This suggests that, for the materials and processing conditions used, the increase in densification as the compaction temperature augments is mainly due to the enhanced plastic deformation of the metallic powder and not to a higher fluidity of the lubricant. It is, however, important to mention that if the lubricant film at the compact/die wall interface has a viscosity which is too low, it might not supply sufficient lubrication during the compaction and ejection processes and lead to a high die wear [4]. Table I also shows that the lubricant properties may have a slight effect on the springback, as suggested by the length variation of the parts after ejection.

Table I : Effect of compaction parameters and mix composition on the density of green samples.

Identification	Lubricant	Compacting	Compacting	Density	Length	Porosity**	Porosity + Lubricant ***
		Pressure	Temperature				
		<i>MPa</i>	<i>°C</i>	<i>g/cm<sup>3</sup></i>	<i>cm</i>	<i>% vol.</i>	<i>% vol.</i>
A	Die Wall	620	25	7.25	3.178	7.8	7.8
C	0.75% LiSt	620	25	7.19	3.182	3.9	9.2
C'	Die wall + 0.75% LiSt	620	25	7.18	3.183		
B	Die Wall	620	150	7.38	3.177	6.1	6.1
D	0.75% LiSt	620	150	7.30	3.177	2.4	7.8
E	0.75% LiSt*	480	150	7.20	3.176	3.7	9.1
F	0.75% EBS	620	150	7.31	3.180	2.3	7.8

\* Compacted at 480 MPa to obtain the density of the samples compacted at 25°C/620 MPa with the same mix.

\*\* %porosity = [1- (Density/Theoretical Density of the mixes)] x 100

\*\*\* %(porosity+lubricant) = 100- %Fe x (“Density”/Iron Theoretical Density) =100-(99.25x Density/7.86)

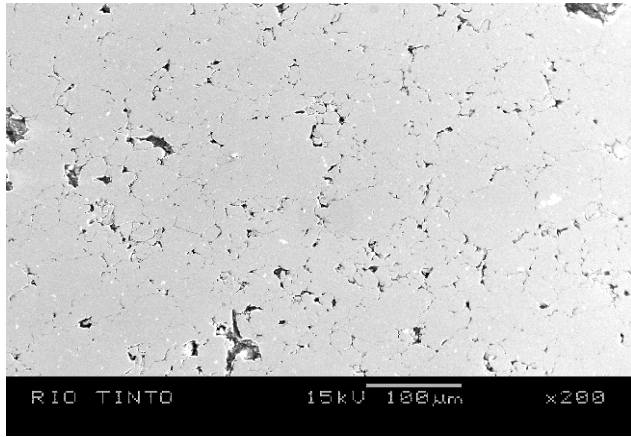
### Porosity distribution

The pore fraction in the specimens can be calculated from the density measurements. Table I shows that a smaller pore fraction is obtained in parts with admixed lubricants. This is due to the fact that a large fraction of the pores are filled with the lubricant. It is also shown in Table I that increasing the compaction temperature reduces the porosity from 7.8% to 6.1% with die wall lubrication and from 3.9% to 2.4% in the compacts containing LiSt. It is however important to point out that the removal of the lubricant during delubrication will leave a network of pores.

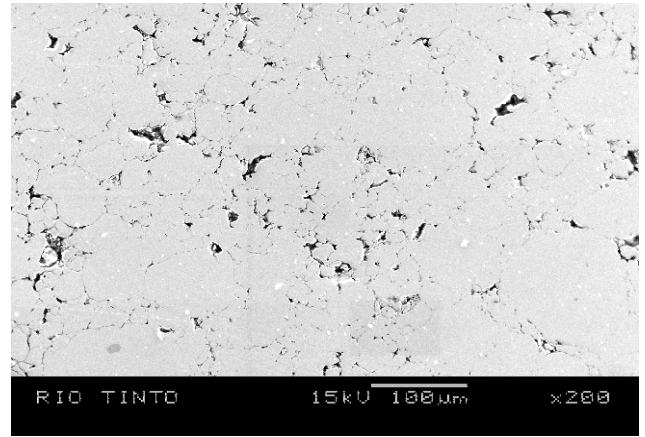
Figure 1 shows the porosity distribution in samples fabricated at 25°C and 150°C with admixed LiSt. The porosity is more important at the edge than at the center of the samples. A similar pattern is seen in specimens compacted with die wall lubrication. However, at 25°C, the differences between the edge and the center are smaller when admixed lubricant is used showing that a more uniform density is achieved. The amount of porosity decreases as the compaction temperature increases. The difference in porosity from the edge to the center is also smaller when the compaction temperature increases, especially for the samples compacted with die wall lubrication.

### Lubricant Distribution

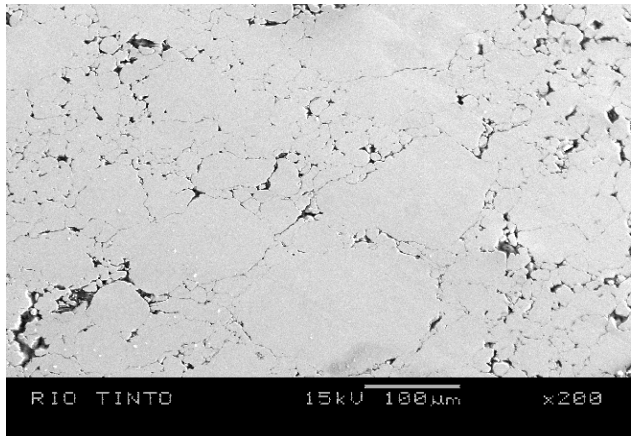
The lubricant distribution was evaluated using WDS and SIMS analytical techniques. In order to evaluate the effect of the sample preparation on the results, preliminary tests were carried out with a pure iron powder specimen compacted at 690 MPa/25°C using die wall lubrication. Figure 2 shows a typical C<sub>3</sub> SIMS image of this specimen. A white network encircling the particles or revealing the presence of pores is visible which indicates that traces of carbon-rich compounds remain in the specimen after rinsing. However, as seen in Table II, the intensity of the signal is weak as compared to the one measured in samples containing 0.6% lubricant, the signal-to-signal ratio being of the order of 300. Therefore, the residual polishing compound should not significantly affect the results of the carbon analyses carried out by either microprobe or SIMS.



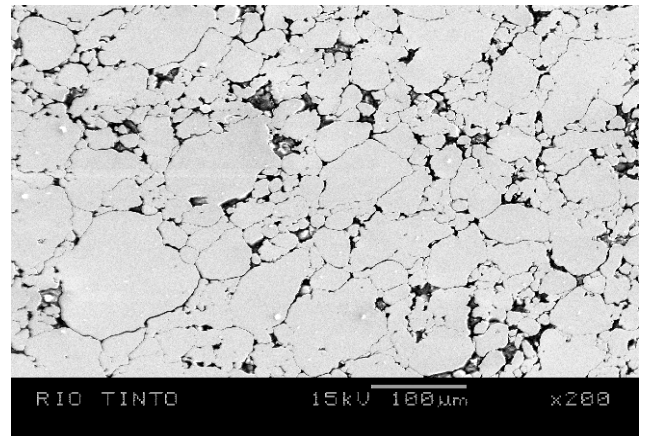
a)



c)



b)



d)

Figure 1 : SEM micrographs of samples compacted at 620 MPa from iron/0.75% LiSt mixes  
a) 150°C, center b) 150°C, edge c) 25°C, center, d) 25°C, edge,.

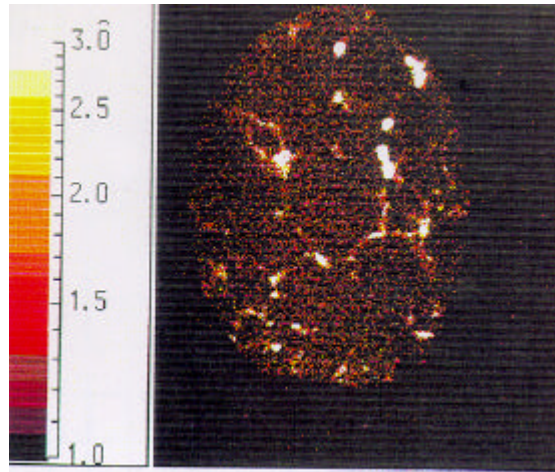


Figure 2 : SIMS carbon image of a polished specimen containing no lubricant.

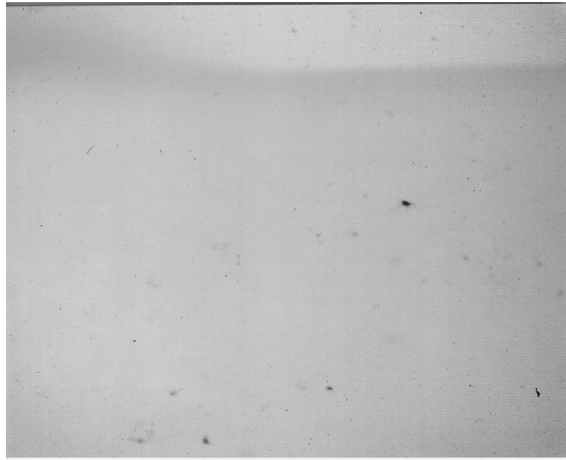
Table II : Comparison of SIMS data for specimens with and without lubricant after metallographic preparation\*.

Specimen Description	Maximum Value Measured (C <sub>3</sub> Counts)
ATOMET 1001 + Die Wall Lubrication	3
ATOMET 1001 + 0.6% LiSt	1 x 10 <sup>3</sup>

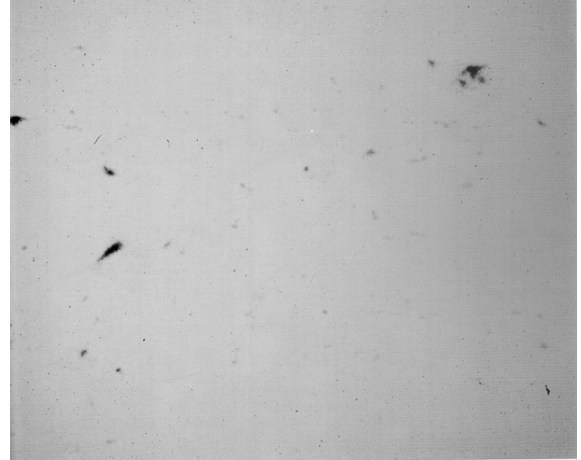
\* Specimens compacted at 690 MPa at 25°C.

Figure 3 presents high-resolution microprobe carbon maps (300X) which show the LiSt distribution in samples compacted at 25°C and 150°C. The following observations can be made:

- At the center of the bars (Figures 3a and 3c), no lubricant is revealed between the particles at either 25 or 150°C, but rather in the pores. More lubricant is detected in the pores of the bars compacted at 25°C.
- At the edge of the bars (Figures 3b and 3d), a stronger carbon signal is obtained as compared to the center. Very little signal comes from the interparticle regions for specimens compacted at 25°C, whereas the contour of the particles appears more delineated by the lubricant in the specimen compacted at 150°C. In both cases, a strong signal which appears more intense in the specimen compacted at 25°C originates from the pores. This may however be partly caused by the larger pore fraction measured in the edge area of the specimen.



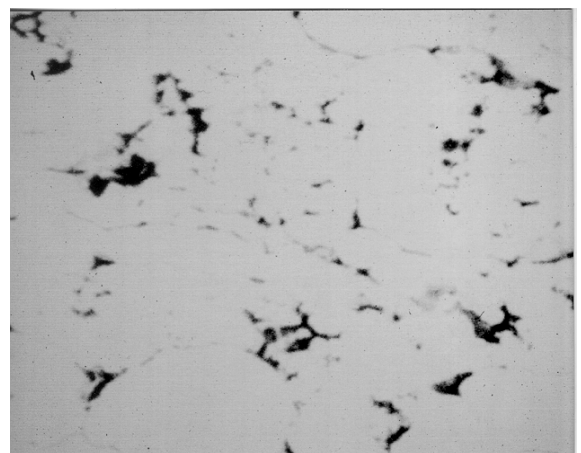
a)



c)



b)



d)

Figure 3 : High-resolution microprobe carbon image of samples containing 0.75% LiSt and compacted at 620 MPa and a)  $T = 150^{\circ}\text{C}$ , center, b)  $T = 150^{\circ}\text{C}$ , edge, c)  $T = 25^{\circ}\text{C}$ , center, d)  $T = 25^{\circ}\text{C}$ , edge. ( The black areas correspond to the carbon signal received by the detector.)



Figure 4 presents the carbon signal obtained along the central axis of the bars with the stage scan technique using an analytical spot size of 80  $\mu$  X 100  $\mu$ . For the specimens compacted at 25°C/620 MPa, a highly irregular carbon distribution profile was obtained (Figure 4a). The peaks observed correspond to pores containing lubricant pockets. For specimens compacted at 150°C/480 MPa (samples having the same density as the samples compacted at 25°C/620 MPa), the same highly irregular carbon distribution profile was obtained (Figure 4b). On the other hand, specimens compacted at 620 MPa/150°C result in lower porosity and in more regular carbon profiles, even if some irregularities can also be observed (Figures 4c and d). The nature of the lubricant (i.e., LiSt vs EBS) is not seen to influence the carbon profile.

In order to more precisely follow the distribution of the lubricant in the samples after compaction, SIMS analyses of lithium were carried out at the center and at the edge of bars containing 0.75% LiSt and compacted at 620 MPa/25°C or 620 MPa/150°C. Five 150  $\mu$  diameter images were acquired in the central and edge regions. Table III lists the average and standard deviation values obtained for each area. The statistical difference between the results was verified using the following statistical test:

$$Z = \frac{\overline{X}_1 - \overline{X}_2}{\sqrt{\frac{s_1^2}{N_1} + \frac{s_2^2}{N_2}}} \quad (\text{eq. 1})$$

where  $\overline{X}_i$  is the mean value of the population i,

$s_i$  is the standard deviation of the population i,

$N_i$  is the number of specimens within the population i.

For a confidence interval of 95%,  $\overline{X}_1$  is different from  $\overline{X}_2$  if  $Z \geq 1.96$ . The calculated "Z" values are listed in Table IV. The small Z value calculated for the part compacted at 25°C indicates that the amount of lubricant present at the center and at the edge of the specimen is statistically similar. It implies that the lubricant does not travel over a large distance when the compaction is carried out at room temperature. On the other hand, the Z value for the part compacted at 150°C suggests that a significantly higher concentration of lubricant is present at the edge of the sample. Therefore, the tendency for the lubricant to be drawn towards the die surface (and the regions of lower density) is increased at higher temperature. This may be explained either by the increased compressibility of the powder mix leading to a higher density, and/or by a higher fluidity of the lubricant at that temperature [3].

The statistical test was also applied to the average values obtained at 25°C and 150°C in both locations. As seen in Table IV,  $Z = 1.47$  for the difference between the lubricant concentrations at the center of the samples compacted at 25°C and 150°C. This small value indicates that the lubricant concentrations at the center of the specimens are similar when compaction is carried out at 25 and 150°C. However, the difference in lubricant concentration after compaction at 25°C and 150°C is higher at the edge of the samples (probability exceeds 99% when the edge values are compared).

**Table III: Results of SIMS for Li analyses (Fe + 0.75% LiSt)**

	620 MPa, 25°C		620 MPa, 150°C	
	Edge	Center	Edge	Center
Mean	11723	11369	16788	9841
Std. Dev.	2835	4117	2367	3399

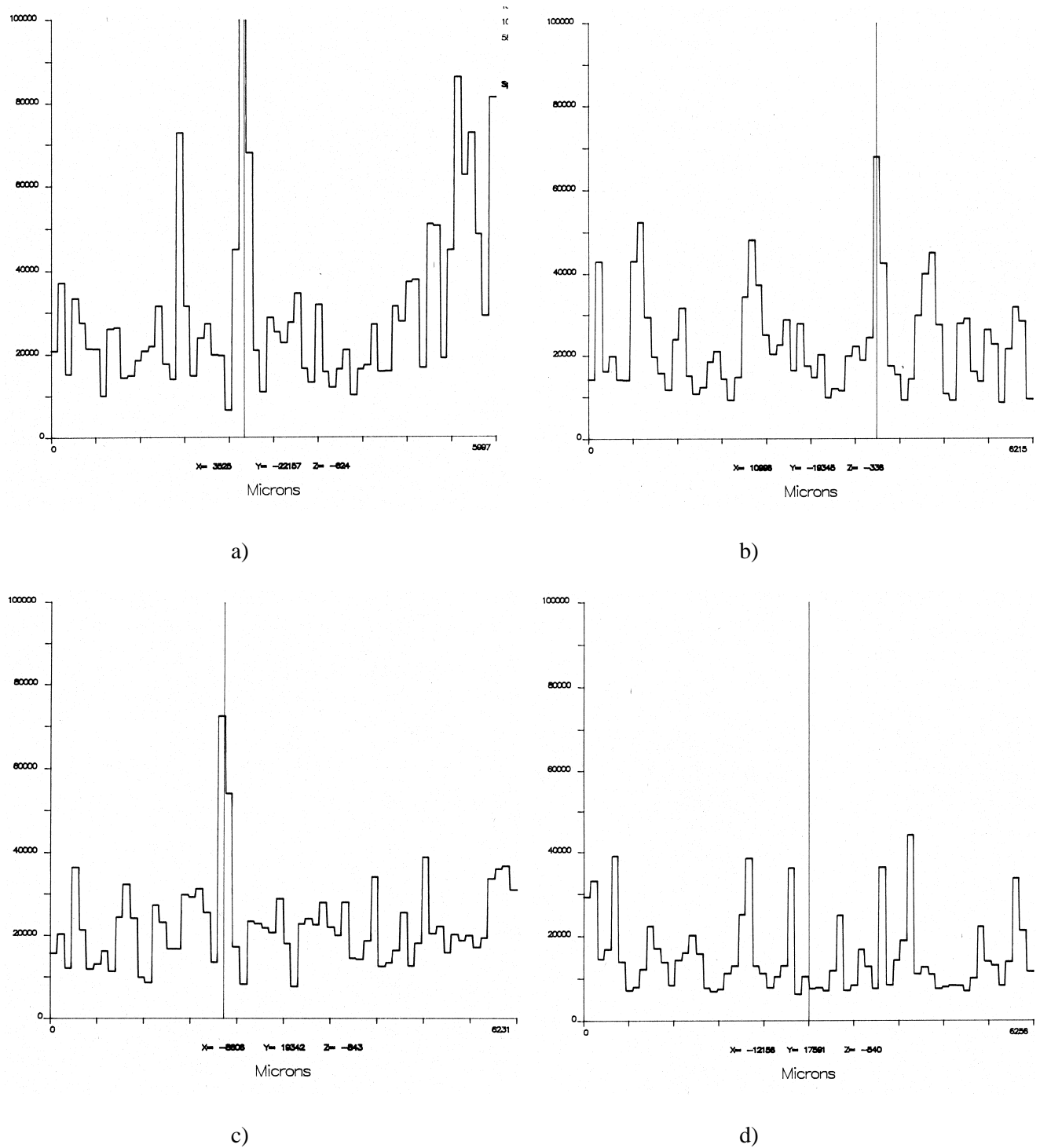


Figure 4 : Carbon distribution profiles from the center ( $d = 0$ ) to the edge on the center cross section of a bar containing 0.75% lubricant compacted at a) 25°C/620 MPa, LiSt, b) 150°C/480 MPa, LiSt, c) 150°C/620 MPa, LiSt, d) 150°C/620 MPa, EBS.

**Table IV:** Statistical tests on SIMS results.

Values Tested	Z (eq. 1)
25°C edge vs 25°C center	0.27
150°C edge vs 150°C center	6.37
25°C center vs 150°C center	1.47
25°C edge vs 150°C edge	7.26

A second series of SIMS measurements were carried out to determine if the higher tendency for the lubricant to be drawn towards the die surface at 150°C was due to the higher compressibility of the powder mix leading to lower porosity at this temperature or to the higher fluidity of the lubricant at that temperature. Table V presents SIMS measurements obtained on parts compacted to the same final density of ~ 7.20 g/cc achieved either by compacting at 620 MPa/25°C or 480 MPa/150°C. Note that these measurements are based on analyzed areas about 12% larger than those reported in Table III and then could not be directly compared with them. The measurements taken on the edge of the bar compacted at 480 MPa/150°C were erratic as shown by the large standard deviation calculated. The application of the statistical test to compare the edge versus the center data within each bar or the corresponding regions of the bars compacted at 25°C and 150°C failed to identify a significant difference between the results as Z was always smaller than the 80% confidence level. Therefore, for a given density, the system studied and the processing conditions used, the lubricant distribution is not strongly dependent on the compacting route followed. The higher amount of LiSt expelled towards the edges of the compact seems to be mainly related to the higher compressibility of the powder mix and not to the higher fluidity of the lubricant at this temperature.

**Table V:** SIMS results for bars pressed at 7.20 g/cm<sup>3</sup>.

Compacting Parameters	620 MPa, 25°C		480 MPa, 150°C	
	Edge	Center	Edge	Center
Mean	19243	16099	14896	17123
Std. Dev.	2367	1147	6200	1180

## Lubricant Distribution on the Top and Sliding Surfaces of the Compacts

The examination of the sample surfaces provides an indication of the superficial distribution of the lubricant after compaction. A view of the top surface of a sample containing 0.75 wt% LiSt compacted at 25°C/620 MPa is presented in Figure 5a. The dark mounds observed on the sample surface are probably accumulations of LiSt in pores opening at the surface. It is, however, difficult to determine if a thin film of lubricant covers the surface of the samples besides these lubricant mounds.

When the compaction temperature increases to 150°C, the LiSt lubricant is more uniformly distributed on the top surface of the samples (Figure 5b). This may be attributed to the higher deformability of the lubricant itself and to the higher compressibility of the metallic powder leading to smaller pores in the compact. A similar lubricant distribution was observed on the top surfaces of the samples compacted at 150°C with EBS, even if this wax is liquid at that temperature. From the experiments performed in this study, it is difficult to quantify the thickness of the lubricant film covering the surface of the compacts.

The observation of the lateral sliding surfaces shows that a lubricating film composed of superimposed flakes covers the surface of the samples containing either LiSt or EBS compacted at 150°C (Figure 6a). These flakes form during ejection by the stretching of the lubricant expelled on the die wall during compaction. Figure 6b shows that the flakes can measure up to 1  $\mu$  thick. The lubricating film may thus be a few microns thick after ejection. It is interesting to note that a 1  $\mu$  thick film at the surface of a sample represents 0.0014 g of lubricant which corresponds to about 1 wt% of total amount of lubricant added. This suggests that most of the lubricant remains in the samples after compaction. The thickness of the lubricant film would be around 0.1 mm if all the lubricant was expelled on the surface of the samples during compaction.

To evaluate the amount of admixed lubricant that migrates to the die walls, one specimen from each set of experimental conditions was slightly polished to remove the lubricant present on the surface and then sintered at 1120°C for 30 min in dissociated ammonia. The weight loss after sintering (Table VI) indicates that although the compaction temperature leads to a higher deformation of the iron particles and an increased fluidity of the lubricant, the major part of the admixed lubricant remains in the compact. The amount of lubricant expelled on the die wall is smaller than 0.09 wt%. These measurements and the SEM observations suggest that the thickness of the lubricating film expelled on the die walls is between 1 and 10  $\mu$  for the mix compositions and the experimental conditions studied. Additional studies should, however, be carried out to verify if there is a significant variation in the lubricating film thickness for the different experimental conditions investigated and how it can affect the lubrication behavior.

Table VI : Weight loss after sintering.

Identification	Lubricant	Compacting Pressure <i>Mpa</i>	Compacting Temperature <i>°C</i>	Weight loss <i>wt%</i>
A	Die Wall	620	25	0.03
C	0.75%wt LiSt	620	25	0.69
B	Die Wall	620	150	0.03
D	0.75%wt LiSt	620	150	0.69
E	0.75%wt LiSt*	480	150	0.69
F	0.75%wt EBS	620	150	0.71

\*Compacted at 480 MPa to obtain the density of the samples compacted at 25°C/620 MPa with the same mix.

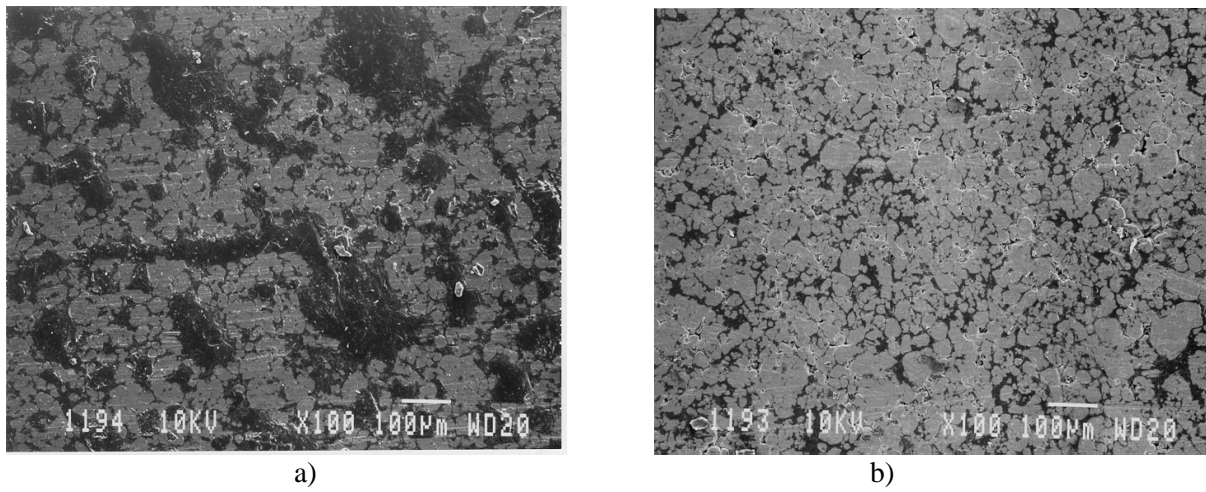


Figure 5: SEM micrographs of the top surface of bars compacted at a) 25°C/620 MPa with LiSt, b) 150°C/620 MPa with LiSt.

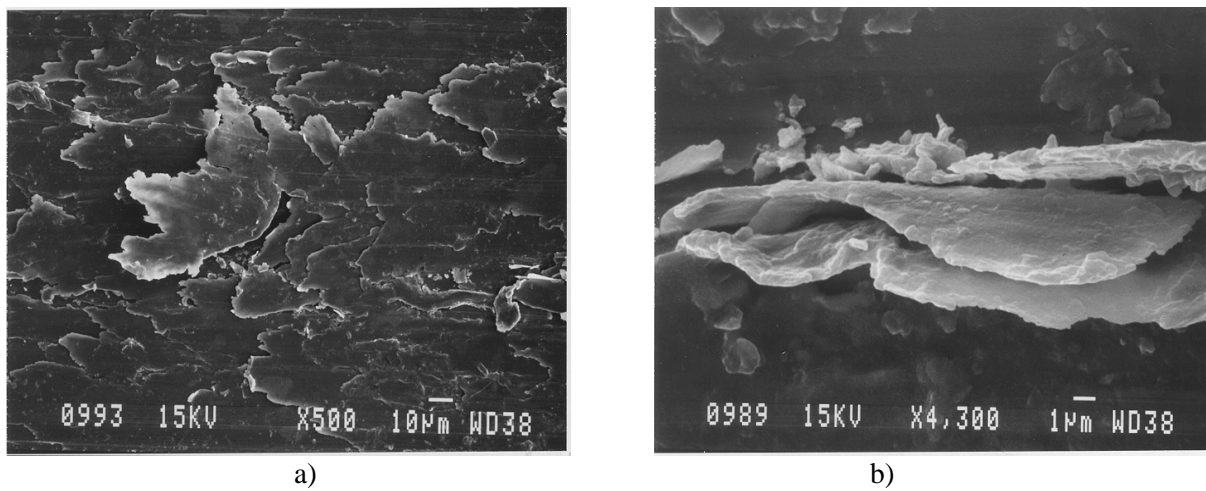


Figure 6 : SEM micrographs of the lateral surface of bars compacted at a) 150°C/620 MPa with LiSt, b) at 150°C/620 MPa with LiSt (details).

## **CONCLUSIONS**

Experiments were conducted to evaluate the effect of compaction temperature on the densification and lubricant distribution after compaction. From the results obtained with iron powder admixed with LiSt and EBS pressed at 25°C and 150°C, the following observations and conclusions can be drawn:

- Higher densification was obtained at higher compaction temperature. The higher compressibility can be related to the enhanced plastic deformability of the metallic powder as the temperature increases. Compared to die wall lubrication, a lower densification was achieved when an admixed lubricant was used. The admixed lubricant might impede pore closure during compaction and reduce the densification of the powder.
- The tendency for the lubricant to flow towards the die surface at higher temperature was observed by characterizing the LiSt lubricant distribution in samples pressed at 25°C and 150°C. However, the higher amount of lubricant expelled towards the edges of the compact was shown to be more related to the higher compressibility of the powder mix and the smaller porosity in the compact rather than to the higher fluidity of the lubricant at 150°C.
- Similar densities and lubricant distributions were obtained for parts compacted at 150°C with LiSt or EBS despite the significantly different viscosity of these two lubricants at that temperature (LiSt is solid while EBS is liquid at 150°C). These results show that the increase in densification as the compaction temperature increases is mainly due to the enhanced plastic deformation of the metallic powder and not to the higher fluidity of the lubricant. However, it is important to mention that, if the lubricant film at the compact/die wall interface has viscosity which is too low, sufficient lubrication might not be ensured during the compaction and ejection processes which will lead to high die wear.
- Looking at the lubricant distribution at the surfaces of the compacted parts, it appears that the compaction temperature (25°C vs 150°C) and pressure (480 vs 620 MPa) do not strongly affect the amount of lubricant expelled on the die walls. Indeed, most of the lubricant (> 93%) remains in the samples after compaction. Additional work should, however, be performed to evaluate the effect of the different experimental conditions on the thickness of the lubricating film on the surface of the samples after compaction. From the weight variation measurements during sintering and the observations of the sample surfaces, it is believed that the lubricant film thickness is about 1 to 10  $\mu$  for the experimental conditions studied.

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