UPDATING THE MAPLUB® GREASES RANGE

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ABSTRACT

MAPLUB® greases have been developed in the 1990s to fulfill the needs of the space industry in the field of mechanism lubrication. MAPLUB® -a greases were the first range commercialized in 1998. These greases have been updated to MAPLUB® -b range in 2006 to substitute the polytetrafluoroethylene particles included in their formulations due to an obsolescence.

Today we have been updating the greases range to MAPLUB®-c to remove the use of solvent in the production process of the greases. This evolution has been decided due to a future obsolescence on the solvent used until now and linked to the strategy of our company to remove all the solvents in the next years to propose safer products with less-environmental impact.

This paper summarizes the validation tests which have been done so far to characterize the new version of MAPLUB® -c greases. All the properties were compared to the current version of MAPLUB® -b greases and to the former one, MAPLUB® -a range.

1. INTRODUCTION

Since its creation in 1986, MAP has developed numerous products for the space industry: coatings for thermo-optical control for spacecraft, varnishes for protection of printed circuit boards (PCB) and electronic components, adhesives, antistatic coatings for launchers and lubricants for satellites mechanisms.

The first lubricants[1], MAPLUB®-a range products were commercialized in 1998. This range was composed of four greases. MAPLUB® PF-a greases were formulated based on very low outgassing perfluoropolyether oil (PFPE) and polytetrafluoroethylene (PTFE) particles. Two products were developed the first one MAPLUB® PF 100-a [1] and the second one MAPLUB® PF 101-a in which molybdenum disulfide is added [2].

MAPLUB® SH-a greases were formulated based on low outgassing synthetic hydrocarbon oil and polytetrafluoroethylene particles. Two products were developed: the first one MAPLUB® SH 050-a [3] and the second one MAPLUB® SH 051-a in which molybdenum disulfide is added [4].

Due to the obsolescence in the PTFE particles used for the formulation of MAPLUB®-a greases, new versions of these greases were developed based on a new reference of PTFE particles. This range was called MAPLUB®-b and commercialized in 2006.

Four greases formulations were updated using this new PTFE particles. The references were kept the same:

- MAPLUB® PF 100-b [5] substituting MAPLUB® PF 100-a;
- MAPLUB® PF 101-b [6] substituting MAPLUB® PF 101-a;
- MAPLUB® SH 100-b [7] substituting MAPLUB® SH 100-a;
- MAPLUB® SH 101-b [8] substituting MAPLUB® SH 101-a.

Until now, the production of the MAPLUB®-a and MAPLUB®-b greases used several solvents. The strategy of MAP is to avoid any solvents from the production process and the products formulation in the next years.

It was then decided to remove the use of solvents and to drastically change the way of producing the greases. The challenge was to use the same raw materials to keep the properties of the greases as the same.

This work has been done in the frame of two Research and Technology (R&T) contracts financially supported by CNES [9, 10, 11, 12]. This work has been divided in two parts, the first one was dedicated to the numerical simulations of the greases to improve the understanding of their behaviour in the mechanisms [13, 14].

The first part of this work has been conducted by LaMCoS (Laboratoire de Mécanique des Contacts et des Structures-INSA Lyon-France) and CNES on several greases used in space (MAPLUB® and its competitors),

[1] A first range of lubricants was designed and commercialized in 1996: MAPLUB® PF250, MAPLUB® PF251, MAPLUB® SH100 and MAPLUB® SH1 101. These products were substituted by MAPLUB® -a greases in 1998.

whose base oils are either polycyclopentane or perfluoroether and whose thickener is PTFE powders.
This study shows a typical behavior of these oils, i.e.: high torques at low speeds (especially at starting)[2]
Due to fine PTFE particles that enter the contact and lining the surfaces, these MAPLUB®-b greases appear to be better in terms of contact and friction (Surface protection, friction coefficients, contact lifetime…). It is then recommended to study this specific behavior during the design studies of the mechanisms: motorization margins, kinematics, speeds…

The second part of this work was dedicated to the production process updating and the characterization of the properties of the new version of MAPLUB® -c greases. As the MAPLUB® -c greases were formulated using the same raw materials used for MAPLUB® -a and MAPLUB® -b greases, it is then expected a same tribological behavior, i.e.: high torques at low speed. This is a normal phenomenon which must be managed using margin motorization. The lifetime of the lubricated components is not affected by such behavior.
This paper first presents the properties of the new version of MAPLUB®-c greases. Finally, these properties are compared to those of the current version MAPLUB®-b and to the former one, MAPLUB® -a range.

2. MATERIALS, PROCESSES AND TECHNIQUES

2.1. Materials

MAPLUB® PF greases are formulated based on very low outgassing perfluoropolyether oil (PFPE) and polytetrafluoroethylene particles for MAPLUB® PF 100-b and MAPLUB® PF 101-b. MAPLUB® PF 101-b contains additional molybdenum disulfide.

For MAPLUB® SH, these greases are formulated based on low outgassing synthetic hydrocarbon oil and polytetrafluoroethylene particles: MAPLUB® SH 100-b and MAPLUB® SH 101-b. This last reference contains additional molybdenum disulfide.

The new versions of MAPLUB®-c greases are based on the same formulations. It was decided to keep the characteristics and the heritage of MAPLUB®-b greases which have been used on numerous satellite programs since 2006.

2.2. Processes

Initially, solvents were used in the production process of the MAPLUB® greases to improve the mixing between solid particles of PTFE and oils. Moreover, quite high concentration of PTFE is foreseen to keep as low as possible oil separation. Due to the very different properties of the oils used in MAPLUB® PF and MAPLUB® SH, two different solvents were used.

To overcome the complete removal of solvent, dedicated production equipment's have been identified to mix oil with solid particles. These devices are generally used to produce formulations with high loading particles such as adhesives, mastics, concrete, or specific greases.

2.3. Techniques

Outgassing rates are measured further to ECSS-Q-ST-70-02C standard [15]. The measurements were performed at Airbus Stevenage facility.
Some rheological properties such as viscosity were measured in-house by MAP further to the following ISO 3219 standard which are included in the reference section [16]. This standard has been adapted to the viscosity measurement of such greases.
All the other properties were measured at INS site further to the following ISO and ASTM standards which are included in the reference section:

- Worked penetration and unworked penetration, 60 strokes on Normalab Analis penetrometer further to NF ISO 2137/ASTM D 217 at 25°C [17];
- Oil separation further to ASTM D6184 using a conical sieve put during 30h at 100°C [18];
- Drop points measurements further to the ASTM D566 standard [19];
- Evaporation loss further to ASTM D972 standard measured at 121°C during 24h [20];
- Viscosity at 40°C and 100°C on the oils using a kinematic viscosimeter SVM 3001 from Anton

[2] The behavior of MAPLUB®-b in low-speed ball bearings (torque peak generation) is "normal". It can be observed with other greases which are suspensions of particles in an oil, such as: MAPLUB®-a, Braycote 601, Lubcon Ultratherm… The phenomena at the origin of the torque peaks in the low-speed ball bearings are complex and come from the competition of accommodation of the speeds in the surface or in the volume

Paar further to ASTM D7042 standard [21]. The viscosity index has been measured further to the D2270 standard [22].

3. QUALIFICATION PLAN

To qualify the new version of MAPLUB® greases, their characteristics must meet the requirements listed in Tabs.1, 2 and 3. These requirements come from the characteristics of the current MAPLUB® -b greases and from the ECSS-Q-ST-70-02C outgassing standard [15].
As the composition of the MAPLUB® -c remained unchanged, only the production process was updated, it was decided to carry out a partial qualification to validate this change.

Table 1. Requirements for MAPLUB® -c greases

Properties	Requirements
RML (%)	≤ 1
CVCM (%)	< 0.1

Table 2. Requirements for MAPLUB® PF-c greases at 20°C

	Requirements	
Properties	MAPLUB® PF100-c	MAPLUB® PF101-c
Viscosity (Pa.s) at 10 s^{-1}	44.0 ± 7.0	44.0 ± 7.0
Viscosity (Pa.s) at 100 s^{-1}	8.3 ± 2.0	8.3 ± 2.0

Table 3. Requirements for MAPLUB® SH-c greases at 20°C

	Requirements	
Properties	MAPLUB® SH100-c	MAPLUB® SH101-c
Viscosity (Pa.s) at 10 s^{-1}	35.0 ± 7.0	42.0 ± 7.0
Viscosity (Pa.s) at 100 s^{-1}	5.0 ± 2.0	5.8 ± 2.0

4. RESULTS

4.1. Outgassing data

The outgassing properties were measured at the Airbus Stevenage facility. The results are listed in Tab.4 [23, 24]. All the results were compliant with the ECSS-Q-ST-70-02C [15].

Table 4. Outgassing results for MAPLUB® -c greases: MAPLUB® PF100-c and MAPLUB® SH100-c [23] and MAPLUB® PF 101-c and MAPLUB® SH 101-c [24]

MAPLUB® -c version Batch	TML (%)	RML (%)	CVCM (%)
MAPLUB® PF 100-c R.14.19.15.01	0.09	0.09	0.05
MAPLUB® PF 101-c R.14.19.17.01	0.07	0.06	0.02
MAPLUB® SH100-c R.14.19.16.01	0.14	0.14	0.05
MAPLUB® SH 101-c R.14.19.18.01	0.20	0.19	0.06

4.2. Rheological properties

The values of the viscosity measurements are listed in Tabs.5 and 6. The results are an average of seven productions' batches.
All the results were compliant with the requirements (Tables 2 and 3).

Table 5. Viscosity measurements for MAPLUB® PF-c greases at 20°C

Properties	MAPLUB® PF100-c	MAPLUB® PF101-c
Viscosity (Pa.s) at 10 s^{-1}	42.2 ± 1.9	41.2 ± 0.5
Viscosity (Pa.s) at 100 s^{-1}	7.9 ± 0.2	8.2 ± 0.2

Table 6. Viscosity measurements for MAPLUB® SH-c greases at 20°C

Properties	MAPLUB® SH100-c	MAPLUB® SH101-c
Viscosity (Pa.s) at 10 s^{-1}	32.3 ± 1.3	37.0 ± 0.1
Viscosity (Pa.s) at 100 s^{-1}	6.8 ± 0.1	7.9 ± 0.2

4.3. INS measurements

4.3.1. Worked penetration and unworked penetration

The results are presented in Tab.7. These results are an average of three measurements.

Table 7. Worked penetration and unworked penetration on MAPLUB® -c greases measured at 25°C [26, 27]

MAPLUB® -c version	Worked penetration 60 strokes (10^{-4} m)	Unworked penetration (10^{-4} m)	NLGI grade
MAPLUB® PF 100-c	325	325	1
MAPLUB® PF 101-c	322	326	1
MAPLUB® SH100-c	357	346	0 – 1
MAPLUB® SH 101-c	347	344	0 - 1

The 60 strokes worked penetration values remain the same as the values of unworked grease.
The NLGI (National Lubricating Grease Institute) grade of the grease is given from the penetration data worked 60 strokes. A cone penetration value of between 355 and 385 tenths of a millimeter corresponds to an NLGI grade 0. This grease can be qualified as "semi-fluid".
Thus, a cone penetration value of between 310 and 340 tenths of a millimeter corresponds to an NLGI 1 grade. This is a grease that can be described as "very soft".
A grease which cone penetration is in between 340 and 355 tenths of a millimeter could be graded 0, 1 or 0-1.

4.3.2. Oil separation

The tendency to oil separation was carried out according to ASTM D6184 using a conical sieve, with an oven for 30 hours at 100 ° C.
The results are presented in the following table.

Table 8. Oil separation results for MAPLUB® -c greases [26, 27]

MAPLUB® -c version	Oil separation (%)
MAPLUB® PF 100-c	7.4
MAPLUB® PF 101-c	7.7
MAPLUB® SH 100-c	3.9
MAPLUB® SH 101-c	4.1

These results are to be assessed depending on the final application. For example, a relatively high oil separation indicates poor storage stability without oil release, but higher lubrication performance under low load (ease of lubricant accessibility). MAPLUB® SH versions are less sensitive to oil separation.

The order of magnitude is comparable to previous versions, within the limits of repeatability of the measurement.

4.3.3. Drop point

The dropping point measurements are listed in Tab.9. This is the temperature at which grease liquefies. This is a maximum instantaneous use limit, with grease turning into oil beyond this point.

Table 9. Drop points for MAPLUB -c greases [26, 27]

MAPLUB® -c version	Drop point (°C)
MAPLUB® PF 100-c	143
MAPLUB® PF 101-c	158
MAPLUB® SH100-c	> 280
MAPLUB® SH 101-c	224

4.3.4. Evaporation

The mass losses by evaporation were determined at 121 ° C for 24 hours. The results obtained are presented in the table below. Very low evaporation rates were measured, justifying the use of the grease for ultra-high vacuum and space applications.

Table 10. Mass loss for MAPLUB® -c greases [26, 27]

MAPLUB® -c version	Mass loss (%w/w)
MAPLUB® PF 100-c	< 0.01
MAPLUB® PF 101-c	< 0.1
MAPLUB® SH100-c	< 0.01
MAPLUB® SH 101-c	< 0.1

4.3.5. Viscosity of the oils

The results of the viscosity measurements on neat oils are listed in the Table 11.

Table 11. Viscosity measurements of the neat oils [25]

MAPLUB® -c version	Viscosity at 40°C ($mm^2.s^{-1}$)	Viscosity at 100°C ($mm^2.s^{-1}$)
MAPLUB® PF oil	142.0	41.1
MAPLUB® SH oil	105.2	14.0

4.4. Comparison of MAPLUB greases properties

In the following tables we compare the properties of MAPLUB® -c greases to the former ones MAPLUB® -a and MAPLUB® -b.
For MAPLUB® PF 100-c and MAPLUB® PF 101-c (Tabs 12 and 13), the characteristics obtained are quite the same as those measured on MAPLUB® -b greases.
A significant decrease in the evaporation rate has been observed probably due to the new process production in which no solvent is used.

Table 12. Comparison of the properties of MAPLUB® PF100 greases

Properties	MAPLUB® PF 100-a	MAPLUB® PF 100-b	MAPLUB® PF 100-c
Worked penetration 60 strokes (10^{-4} m)	269	320	325
Unworked penetration (10^{-4} m)	288	315	325
NLGI grade	2	1	1
Density	1.9	1.93	1.92
Viscosity (Pa.s) at 10 s^{-1}	69	44	42.2
Viscosity (Pa.s) at 100 s^{-1}	12	8.3	7.9
Oil separation	2.65	6.1	7.4
Evaporation loss	0.17	0.9	< 0.01

Table 13. Comparison of the properties of MAPLUB® PF101 greases

Properties	MAPLUB® PF 101-a	MAPLUB® PF 101-b	MAPLUB® PF 101-c
Worked penetration 60 strokes (10^{-4} m)	281	311	322
Unworked penetration (10^{-4} m)	289	302	326
NLGI grade	2	1	1
Density	1.9	1.95	1.95
Viscosity (Pa.s) at 10 s^{-1}	69	44	41.2
Viscosity (Pa.s) at 100 s^{-1}	13	8.3	8.2
Oil separation	3.26	4.7	7.7
Evaporation loss	0.37	1.1	< 0.1

For MAPLUB® SH 100-c and MAPLUB® SH 101-c (Tabs 14 and 15), the characteristics obtained are quite the same as those measured on MAPLUB® -b greases, except the worked and unworked penetration which are closer to those of MAPLUB® -a range.
The NLGI grade for MAPLUB® -c greases (0-1) is in between those of MAPLUB® SH-a (0-1 or 0) and MAPLUB® SH-b (1) greases.

Table 14. Comparison of the properties of MAPLUB® SH 100 greases

Properties	MAPLUB® SH 050-a	MAPLUB® SH 100-b	MAPLUB® SH 100-c
Worked penetration 60 strokes (10^{-4} m)	348	329	357
Unworked penetration (10^{-4} m)	335	326	346
NLGI grade	0-1	1	0-1
Density	1	1.08	1.16
Viscosity (Pa.s) at 10 s^{-1}	26	35	32.3
Viscosity (Pa.s) at 100 s^{-1}	4	5	6.8
Oil separation	4.19	3.2	3.9
Evaporation loss	0.72	0.2	< 0.01

Table 15. Comparison of the properties of MAPLUB® SH 101 greases

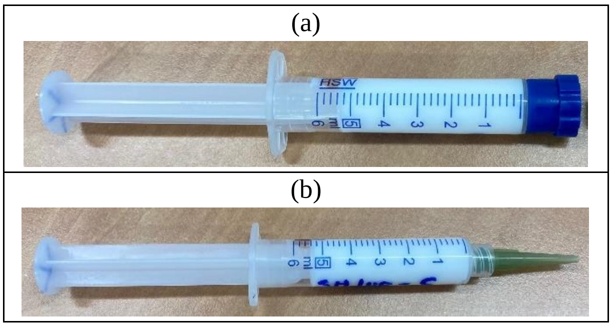
Properties	MAPLUB® SH 051-a	MAPLUB® SH 101-b	MAPLUB® SH 101-c
Worked penetration 60 strokes (10^{-4} m)	365	319	347
Unworked penetration (10^{-4} m)	248	313	344
NLGI grade	0	1	0-1
Density	1	1.1	1.2
Viscosity (Pa.s) at 10 s^{-1}	22	42	37.0
Viscosity (Pa.s) at 100 s^{-1}	4	5.8	7.9
Oil separation	3.82	2.0	4.1
Evaporation loss	0.22	0.2	< 0.1

All the characteristics of MAPLUB® -c greases are described in their TDS: MAPLUB® PF 100-c [28], MAPLUB® PF 101-b [29], MAPLUB® SH100-b [30] and MAPLUB® SH101-b [31].

4.5. Improvement of the easiness of use

To improve the easiness of use of these lubricants, they have been packaged in 5 mL syringes (Fig.1a). The grease is then easily applied directly in the mechanisms using a needle (Fig.1b).

Figure 1. Syringes filled with MAPLUB® SH100-c grease. (a) Syringe with cap and (b) syringe with application needle



All the greases MAPLUB® PF 100-c, MAPLUB® PF 101-c, MAPLUB® SH 100-c, and MAPLUB® SH 101-c have been successfully stored at room temperature in syringes without significant change in rheological characteristics.
All the MAPLUB-c greases are now available in pack of 10 syringes of 5 mL with dedicated needles (plastic and steel). This new packaging replaces the pots used until now.

5. CONCLUSION

To remove the use of solvents in MAPLUB® -b greases production process and to continue the production and the commercialization of greases for space uses, a new solvent-free production process has been developed. The new version of MAPLUB® greases called MAPLUB® -c range has been developed and characterized. The formulation of the products is based on the same raw materials as previously used for MAPLUB® -b greases.
A comparison of the properties of the 3 ranges MAPLUB® -a, MAPLUB® -b and MAPLUB® -c has

been done. All the results obtained meet the requirements. The new version MAPLUB® -c packaged in syringes are commercialized from 2022.

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