EVALUATION OF A MODIFIED PURIFICATION PROCESS FOR MAPSIL® 210

G. Sierra[(1)], D. Sacramento[(1)], S. Solé[(1)], O. Guillaumon[(1)]

[(1)] MAP, ZI - 2 Rue Clément Ader, 09100 Pamiers, France, g.sierra@map-coatings.com

ABSTRACT

MAPSIL® 210 is a thermal grease which was developed in the 1980s using the liquid-liquid purification process developed by CNES.

In order to remove the use of organic solvents included in the purification process and to prevent a potential breakdown in the supply chain with regard to our customers, the production process of MAPSIL® 210 has been updated.

This paper summarizes the validation tests which have been done so far to characterize the new version of MAPSIL® 210. All the properties that were controlled met the requirements and were identical to the current version of MAPSIL® 210.

1. INTRODUCTION

Since its creation in 1986, MAP has developed numerous products for the space industry. The essential amount of these products consists of silicone-based greases, adhesives, varnishes or coatings.
MAPSIL® 210 is a silicone thermal grease obtained thanks to a liquid-liquid purification process (CNES patent) which makes it possible to obtain degassing values compatible with space applications [1]. In order to remove the use of organic solvents that do not comply with new European environmental regulations (REACH), the purification process was modified.
Moreover, to anticipate a potential breakdown in the supply chain which would lead to space qualified products breakage, we decided to synthesize in-house the silicone polymers initially used for the production of MAPSIL® 210.
In order to check the properties of the new version of MAPSIL® 210, we defined the following qualification plan:

1. Control of the product at initial stage;
2. Application and characterization;
3. Comparison of the properties of the new version of MAPSIL® 210 with the current one.

Firstly, this paper presents the properties of the current version of MAPSIL® 210. Secondly, these properties are compared with those of the new version of MAPSIL® 210.

2. MATERIALS, PROCESSES AND TECHNIQUES

2.1. Materials

MAPSIL® 210 is a thermal grease whose composition is based on the mixture of a silicone polymer, metallic oxides and additives. It is a single component which remains liquid. The characteristics of the thermal grease [2, 3] are listed in Tab.1.

Table 1. General properties of MAPSIL® 210 thermal grease

	MAPSIL® 210
Solids content (%)	100
Density	2.3
Viscosity at 50 s^{-1} (Pa.s)	98
TML (%)	0.05
RML (%)	0.04
CVCM (%)	0.04
Thermal conductivity ($W.m^{-1}.K^{-1}$)	0.4

2.2. Techniques

Outgassing rates are measured further to ECSS-Q-ST-70-02C standard [1]. The measurements were performed at Airbus Toulouse. Thermal conductivity is measured using the hot-wire method. This method is adapted to liquids and gases thermal conductivity measurement [4-6]. The measurements were performed by LIMATB laboratory.
All the other characteristics were measured in-house by MAP further to the following ISO standards:

- Solids content [7];
- Density using a pycnometer [8];
- Viscosity using RS1 rheometer, Thermofisher [9].

3. QUALIFICATION PLAN

In order to qualify the new version of MAPSIL® 210, its characteristics must fulfill the requirements listed in Tab.2. These requirements are derived from the

characteristics of the current MAPSIL® 210 and from the ECSS-Q-ST-70-02C outgassing standard [1].

Table 2. Requirements for MAPSIL® 210 thermal grease

Properties	Requirements
Solids content (%)	100
Density	2.3 ± 0.2
Viscosity at 50 s^{-1} (Pa.s)	98 ± 25
Viscosity at 100 s^{-1} (Pa.s)	166 ± 52
RML (%)	≤ 1
CVCM (%)	< 0.1
Thermal conductivity ($W.m^{-1}.K^{-1}$)	> 0.4

4. RESULTS

The product's density was measured using a pycnometer further to ISO 2811-1 standard [8]. The value measured is 2.4 and is in the range of the requirements (Tab.2). The solids content value was measured according to ISO 3251 standard and is equal to 100%.
The values of viscosity measurements are listed in Tab.3 and are in good agreement with the values of the current product. Standard deviations of viscosity were measured from three batches of the current product and from 4 batches from new MAPSIL® 210.

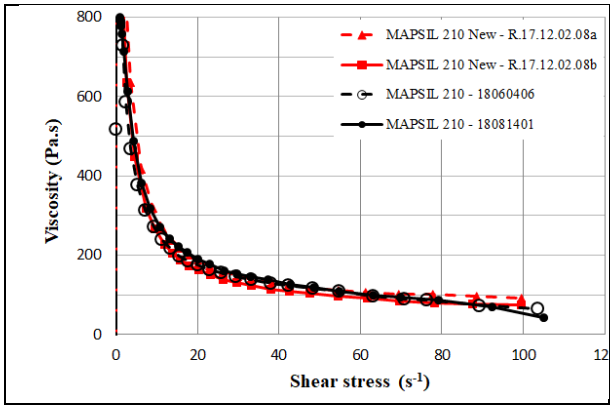
Table 3. Viscosity measurements for MAPSIL® 210 thermal grease

	Viscosity (Pa.s)	
MAPSIL® 210	50 s^{-1}	100 s^{-1}
Current version	98 ± 25	166 ± 52
New version	108 ± 7	177 ± 2

One can observe that the standard deviation of viscosity is lower for the new product compared to the former one. This fact has been attributed to the change in the new product's production process which led into more repeatable properties.

On graph 1, the evolution of viscosity for both products (current and new MAPSIL® 210) is plotted versus shear stress. One can notice the very similar behavior of both products.

Graph 1. Viscosity versus shear stress for current MAPSIL® 210 and new MAPSIL® 210



Once the formulation was defined and then frozen due to the similar properties (solids content, density and viscosity), thermal conductivity was measured using the hot-wire method.
A value of 0.62 $W.m^{-1}.K^{-1}$ was measured for MAPSIL® 210 [10]. This value is a little bit higher than those measured a few years ago. The difference comes probably from the change in the method used for the thermal conductivity measurement.
For the new version of MAPSIL® 210 a value of 0.71 $W.m^{-1}.K^{-1}$ was measured [10]. This value is a little bit higher than those of the former product. It could be attributed to the slight change in metallic oxides ratio.

Table 4. Thermal conductivity of the current and new versions of MAPSIL® 210

MAPSIL® 210	Thermal conductivity ($W.m^{-1}.K^{-1}$)
Current version	0.62
New version	0.71

The outgassing properties were measured at Airbus Toulouse facility. The results are listed in Tab.5 [11].

Table 8. Outgassing results for MAPSIL® 210

MAPSIL® 210	TML (%)	RML (%)	CVCM (%)
Current version	0.05	0.04	0.04
New version	0.05	0.05	0.00

5. FINDING

The current version of MAPSIL® 210 is obtained thanks to a liquid-liquid purification process (CNES patent) which makes it possible to obtain degassing values compatible with space applications [1]. In order to remove the use of organic solvents that do not comply with new European environmental regulations (REACH), the purification process was modified. Moreover, silicon polymers used for the production of MAPSIL® 210 were synthesized in-house.

Solids content, viscosity, density, thermal conductivity and outgassing properties of the new version of MAPSIL® 210 are compliant to the requirements and close to the properties of the current MAPSIL® 210.

6. REFERENCES

1. ECSS-Q-ST-70-02C, Thermal vacuum outgassing test for the screening of space materials
2. Qualification of adhesives and potting resins produced by MAP, CNES report, 89/CT/DRT/TVE/TH n°074, 7 February 1989;
3. MAP, Technical data sheet – MAPSIL 210
4. S.Gauthier Caractérisation thermique de la matière par la méthode 3ω, PhD thesis, Montpellier II University, 2012
5. H. M. Roder. A transient hot wire thermal conductivity apparatus for fluids, Journal of research of the National Bureau of Standards, 86(5) :458480, 1981. 18, 20, 22
6. M. J. Assael, K. D. Antoniadis, and W. A. Wakeham. Historical evolution of the transient hot-wire technique. International journal of thermophysics, 31:1051 - 1072, 2010. 18
7. ISO 3251 standard, Paints, varnishes and plastics – Determination of non-volatile-matter content, 2008
8. ISO2811-1 standard, Paints and varnishes – Determination of density – Part 1: Pycnometer method, 2011
9. ISO 3219 standard, Plastics – Polymers/resins in the liquid state or as emulsions or dispersions – Determination of viscosity using a rotational viscometer with defined shear rate, 1994
10. Measurement of thermal conductivity, LIMATB report, “Rapport MAP_novembre 2018_V1”, November 2018
11. Outgassing report, Airbus M19943.2, 2019