



Séminaire CNES
2022-09-27

NEW HOMOGENEOUS GELLED OILS FOR SPACE MECHANISMS LUBRICATION



POLYMEREXPERT

Putting innovation first

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BACKGROUND AND PURPOSE

Goal of the project:

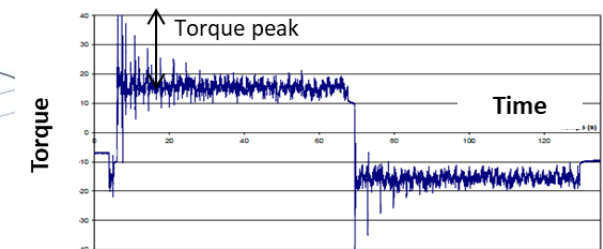
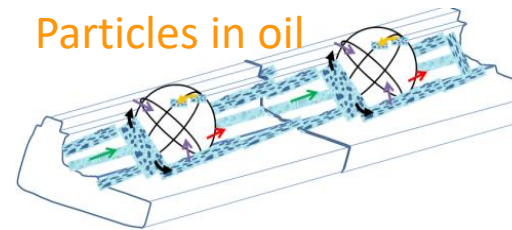
Develop new **homogeneous** gels able to replace PTFE MoS₂-based greases (such as Braycote, Lubcon, Maplub,...)

How to solve these issues ?

- **Homogeneous gels** : no phase separation over time
- **Rheofluidifying properties** : low viscosity of the gels during the rotation of the ball bearings , high viscosity at rest

Drawback of current technology :

Particles in oil



- **failure** of the mechanism due to surface creeping of liquid oil
- **lack of lubricant** in contact over time
- **high torque peaks** when used in ball bearings at low rotation speeds

SPECIFICATIONS



Base oils

- **NYE2001A**
CycloPentane oil (Hydrocarbon)
- **FOMBLIN Z25**
PerFluoroPolyEther oil (PFPE)



Vacuum compatibility: (ECSS-Q-70-02A standard)

- Vapor pressure $< 10^{-8}$ Torr (20°C)
- TML $< 1\%$
- CVCM $< 0.1\%$
- RML $< 1\%$
- Evaporation rate under 10^{-5} Torr
 $< 10^{-5}$ mg.cm².s⁻¹ (100°C)



stability

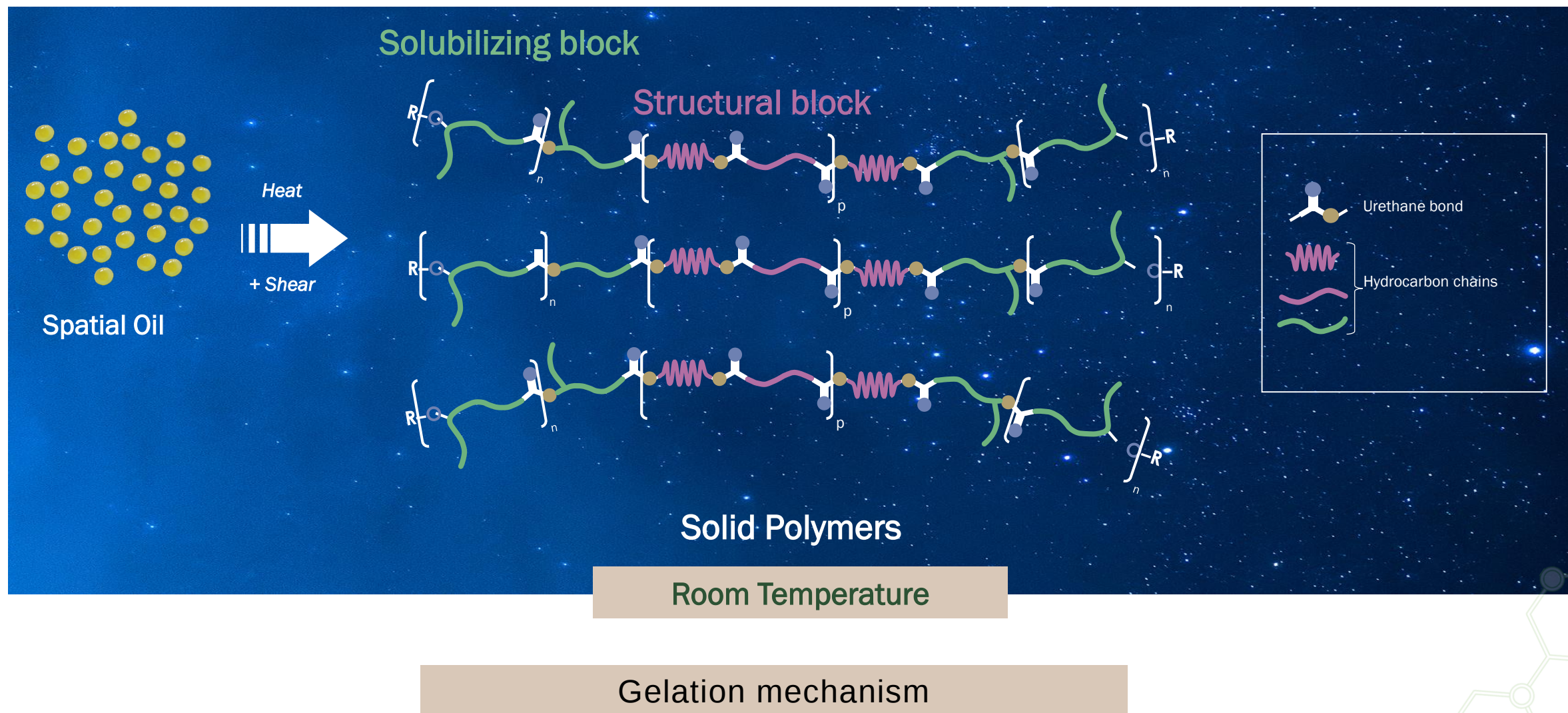
- Constant use :
-120°C $< T <$ +120°C
Occasional use : $< +200^\circ\text{C}$
- Compatibility with metals,
PTFE, Phenolic resins, ...
- Shelf life and in use lifetime :
 > 10 years
- No space radiations
susceptibility



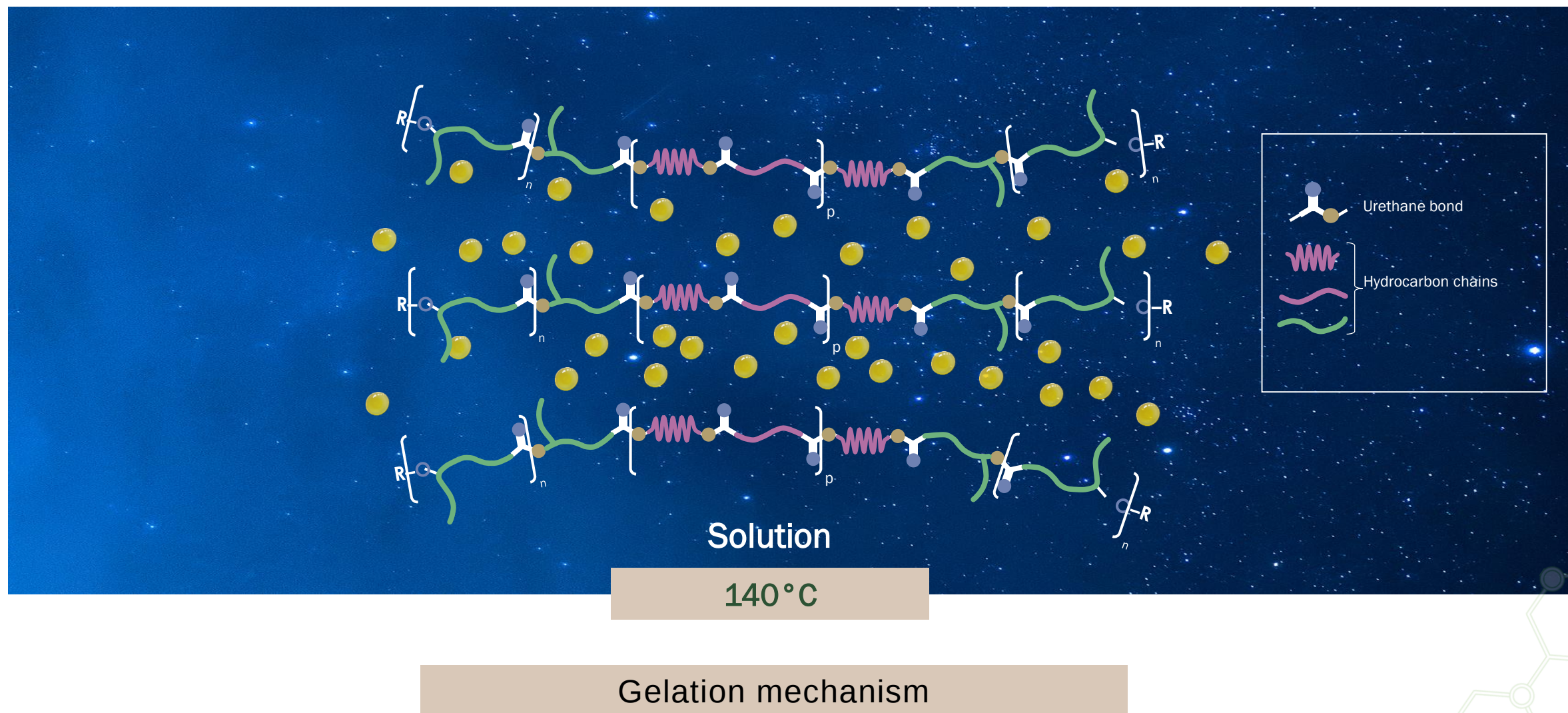
Viscosities (Similar to Maplub SH and PF)

- **In NYE2001A :**
30±10 Pa.s (10s⁻¹) and 7±2 Pa.s (100s⁻¹)
- **In Fomblin Z25 :**
44±10 Pa.s (10s⁻¹)
8±2 Pa.s (100s⁻¹)

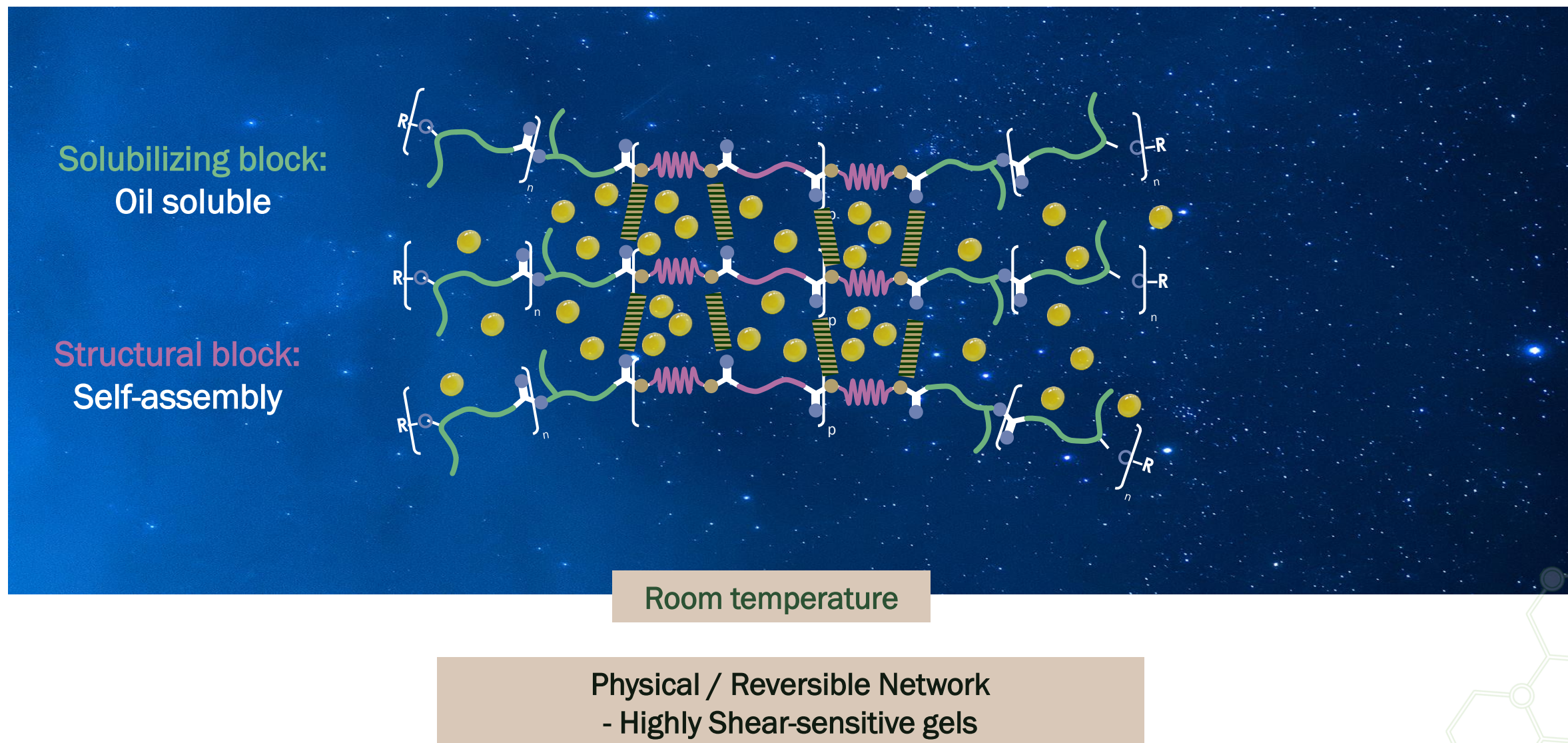
R&D Strategy: self-assembly of supramolecular polymers



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SYNTHESIS OF NEW GELATORS

Synthesis of POLYURETHANE (PU)

01

- Good availability of starting materials

02

- large variety of oil soluble diols and diisocyanates

03

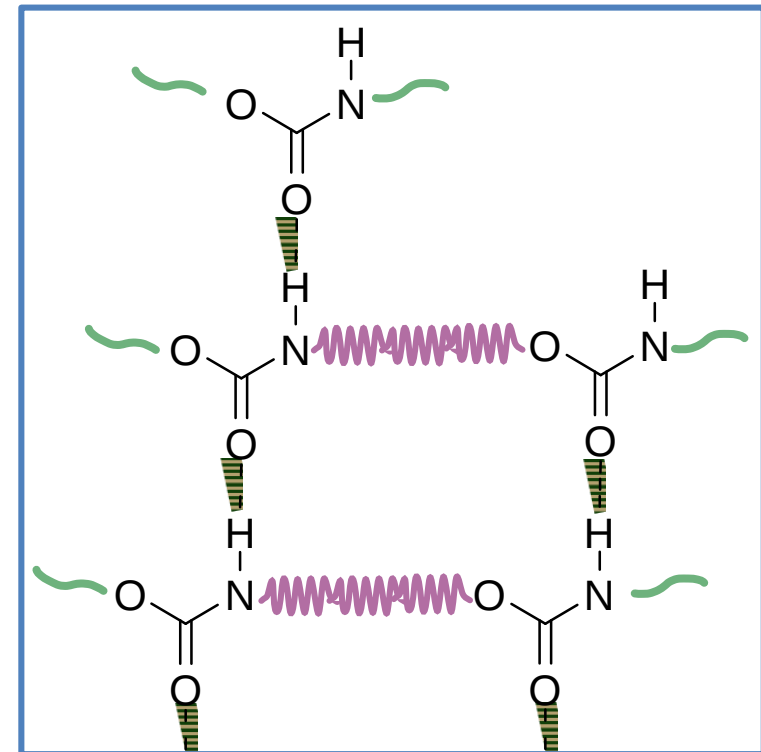
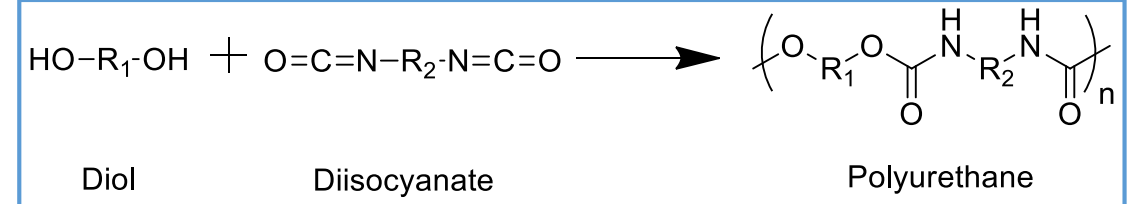
- Good thermal stability

04

- High molecular weight = very low vapor pressure and mass loss

05

- Selection of Solubilizing Block depending on the oil to viscosify



Viscosification of NYE2001A

2 steps synthesis:

Solubilizing block

- functionalization of the polyolefine by a diisocyanate

Structural block

- chain extension using a small molecular weight diol
- termination reaction by an alcohol

Hot solubilization in NYE2001 (IT-DEX20003/IT-DEX20004):

- Concentration : 10 to 15%
- Temperature : 140°C
- Vacuum: 10^{-2} bar
- Solubilization time : 1 to 2 hours

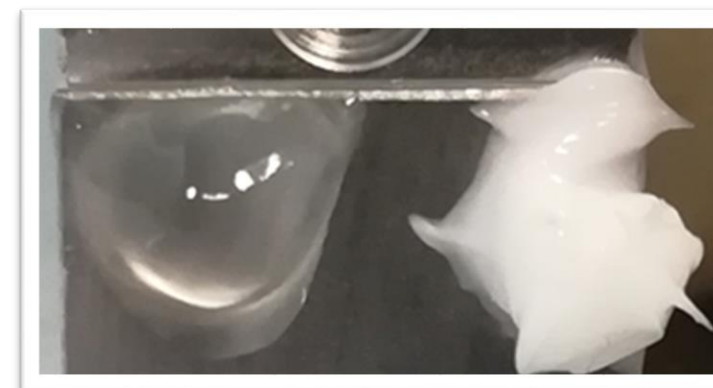
Translucide homogeneous gel

2 Polyurethanes:

PU2000 medium molecular weight

PU3000 high molecular weight

NYE2001A-based gel *MAPLUB SH-100B*



First characterizations of NYE2001A/PU GELS

Medium Mn
PU2000

Key Targets
 $\eta_{10s^{-1}} : 40 \pm 10 \text{ Pa.s}$
 $\eta_{100s^{-1}} : 6 \pm 2 \text{ Pa.s}$
 TML <1%

High Mn
PU3000



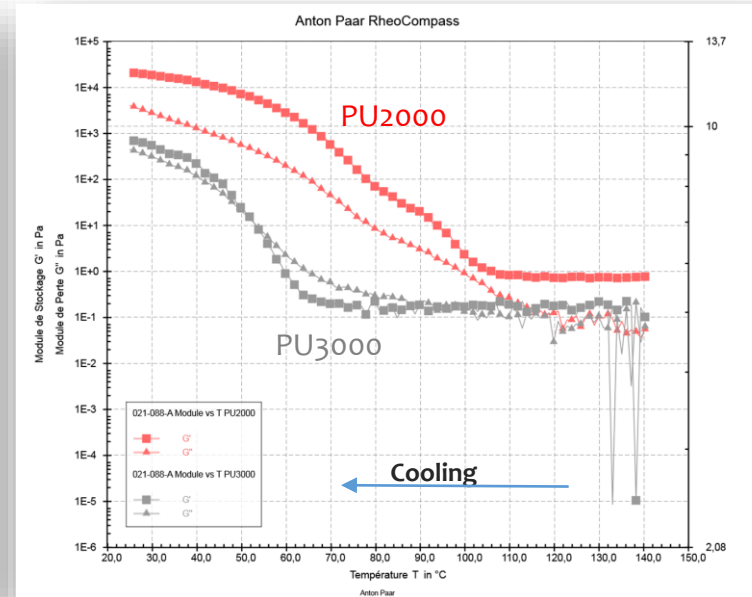
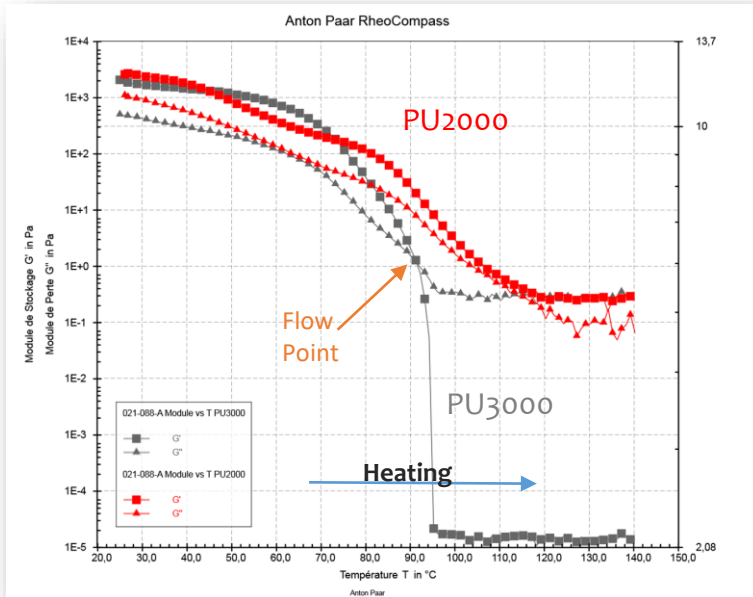
	PU2000 (90 %wt NYE2001A – 10 wt% PU2000)		
	Viscosité 10s ⁻¹	Viscosité 100s ⁻¹	TML
Batch N°1 (150 g)	39 Pa.s	6 Pa.s	99.7 %
Batch N°2 (165 g)	37 Pa.s	6 Pa.s	99,7 %
Batch N°3 (165 g)	41 Pa.s	6 Pa.s	99.5 %



	PU3000 (91 %wt NYE2001A – 9 wt% PU3000)		
	Viscosité 10s ⁻¹	Viscosité 100s ⁻¹	TML
Batch N°1 (160 g)	45 Pa.s	6 Pa.s	99.9 %
Batch N°2 (170 g)	46 Pa.s	7 Pa.s	100 %
Batch N°3 (170 g)	40 Pa.s	6 Pa.s	99.8 %



■ Rheological behavior (temperature)



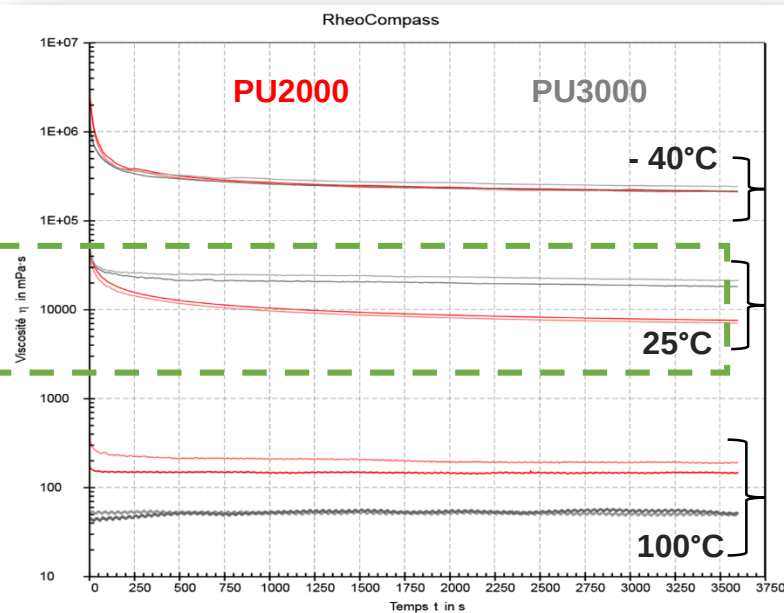
- ✓ Flow point PU2000 > PU3000
- ✓ Structure recovery on cooling for the two gels
 - Slower recovery speed and less "firm" structure (lower G^* modulus) for PU3000

■ Other properties

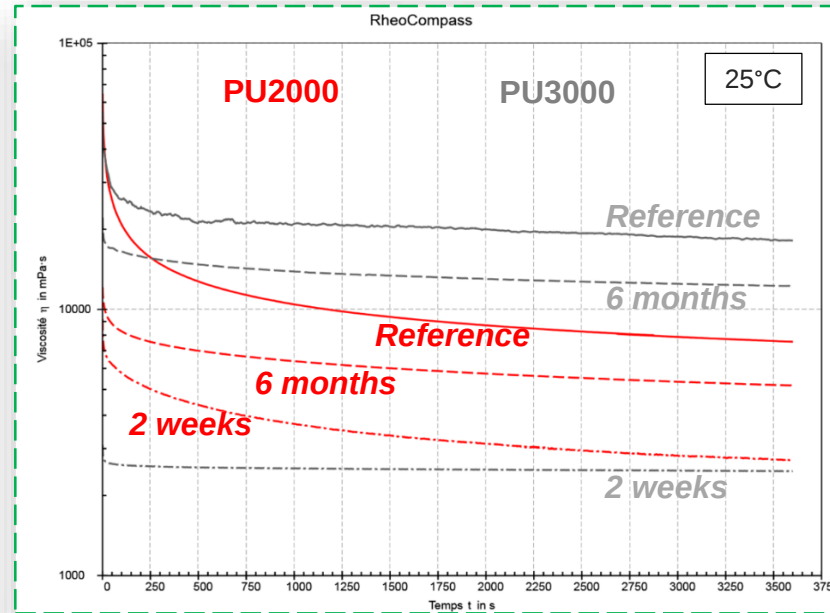
Sample reference	Evaporation loss 22h/121°C (%w/w)	Dropping point(°C)	Apparent density
PU2000 CRG 20160	< 0,1	94	0,83
PU3000 CRG 20161	To be confirmed	88	0,80
Space grease	< 0,1	160-220	< 1

- ✓ Evaporation loss < 0.1% for PU2000 gel, equivalent to space grease
- ✓ Low dropping point compared to a grease → Limited application at high temperature?
- ✓ Relatively low density → Interesting property for mass gain in bearings

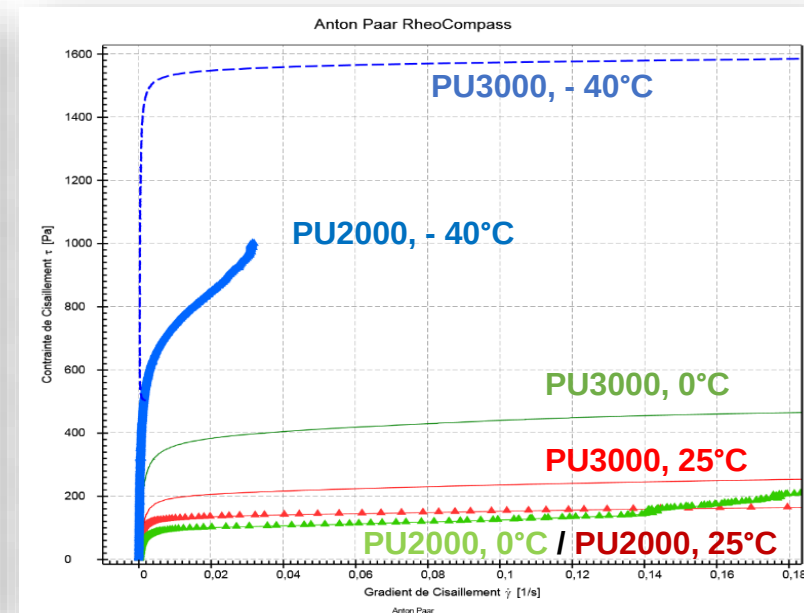
■ Rheological behavior (shear)



- ✓ Dynamic viscosity PU3000 > PU2000 at 25°C but inversely at 100°C (related to flow temperature)
- ✓ Gelled oils are sensitive to shear stress → Shear-thinning behavior for both gels



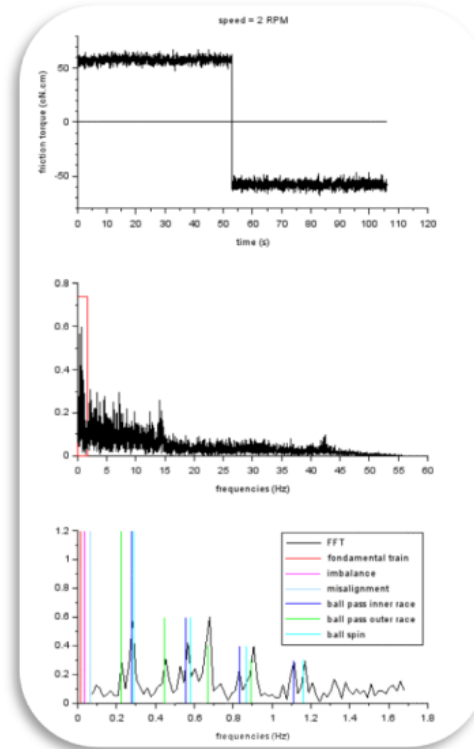
- ✓ Initial dynamic viscosity not restored 6 months after “grease worker” shear whatever the gel → Weak thixotropic effect (too high shear, test for grease)
- ✓ Thixotropy PU2000 > PU3000
- ✓ Shear strength PU2000 > PU3000



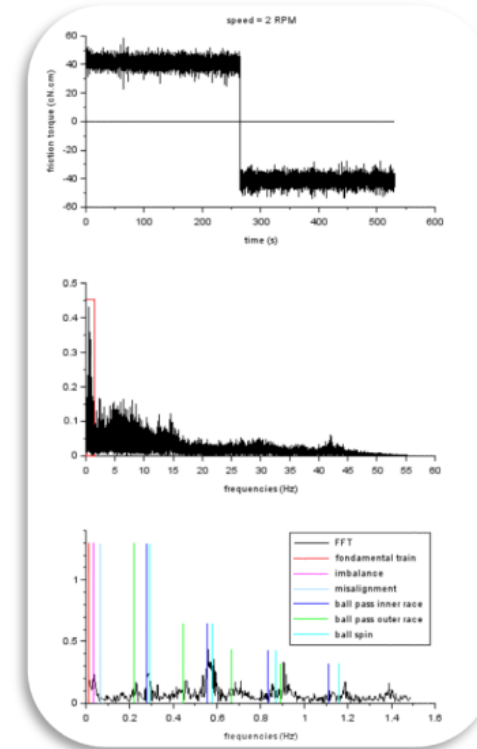
- ✓ Both gels are stress threshold fluids
- ✓ Flow stress PU3000 > PU2000 whatever the temperature (-40°C, 0°C and 25°C)

■ Behavior in a ball bearing* (tests on ADR-ALCEN bench)

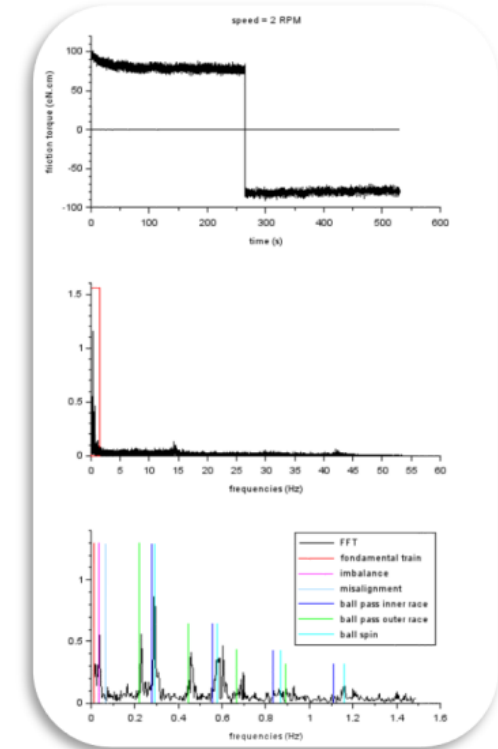
- ✓ Same behaviors for the two gels
- ✓ Low and stable rolling torques (torques twice lower than classical space greases 50 cN.cm instead of 100 cN.cm):
 - Grease behavior for location near the contact
 - Oil behavior for torques
- ✓ Frequency analysis :
 - Same characteristic frequencies but intensity lower for gels, like oil only.
 - Bearing defaults increased by classical space grease (PTFE aggregate ?)



Oil only
Cm = 58 cN.cm, %N = 9.0

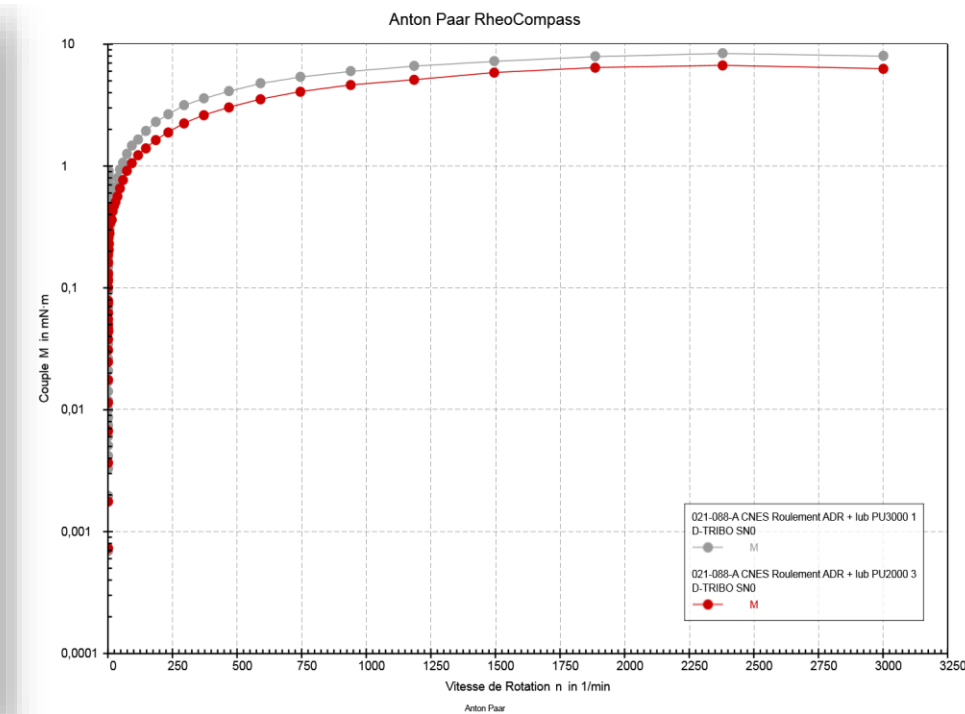
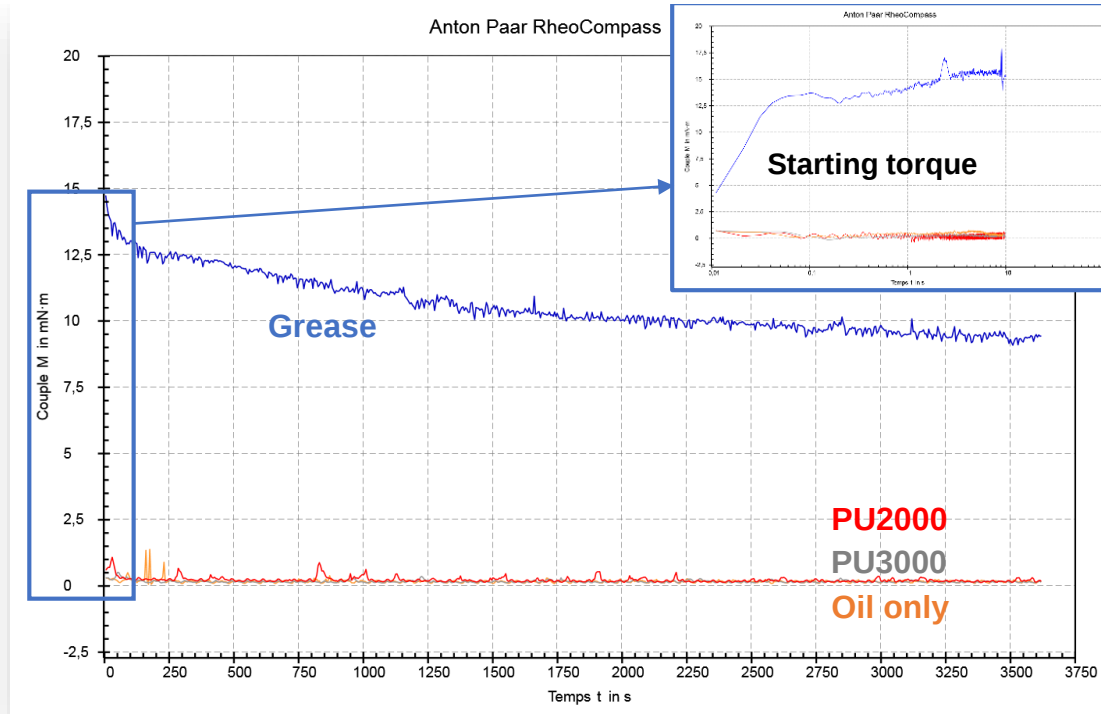
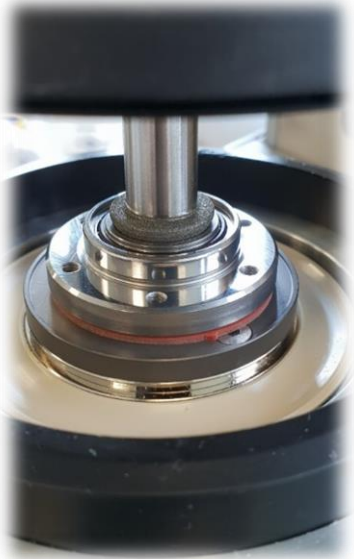


Gelled oil grease
Cm = 41 cN.cm, %N = 14



Classical space grease
Cm = 80 cN.cm, %N = 10

■ Behavior in a ball bearing (InS tests on a rheometer)



- ✓ Same behavior for the 2 gelled oil references, with low rolling torque, identical to the base oil alone and much lower than a grease*
- ✓ Rolling torque stabilization (< 10 mN.m) at high rotation speed
- ✓ Similarity in the range of measured torques between the two test benches (ADR/InS)

TRIBOLOGICAL TESTS ON HV TRIBOMETER

- 3 lubricants :
 - 2 gelled oils : PU2000 & PU3000
 - 1 grease for reference (Maplub pf100-b)
- 1 Hertz contact pressure : 1 GPa

	Kinematics	
	Continuous	Oscillating
Pure sliding	10 mm/s	10 mm/s
	225 mm/s	210 mm/s
Rolling with sliding	10 mm/s ; SRR = 10%	10 mm/s ; SRR = 10%
	125 mm/s ; SRR = 10%	125 mm/s ; SRR = 10%

- 1000 cycles @ 225mm/s, 125 mm/s,
- 100 cycles @ 10 mm/s

- Environment : high vacuum 4.10^{-7} mbar

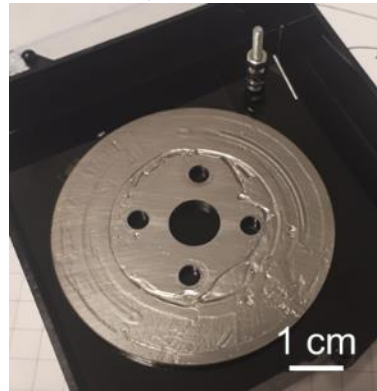


TRIBOLOGICAL TESTS ON HV TRIBOMETER



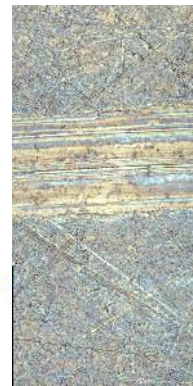
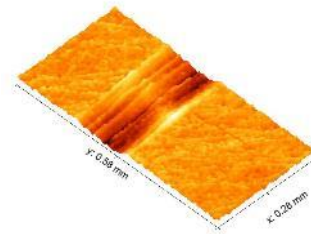
▲ Calibrated thickness of 100 μm

Friction test



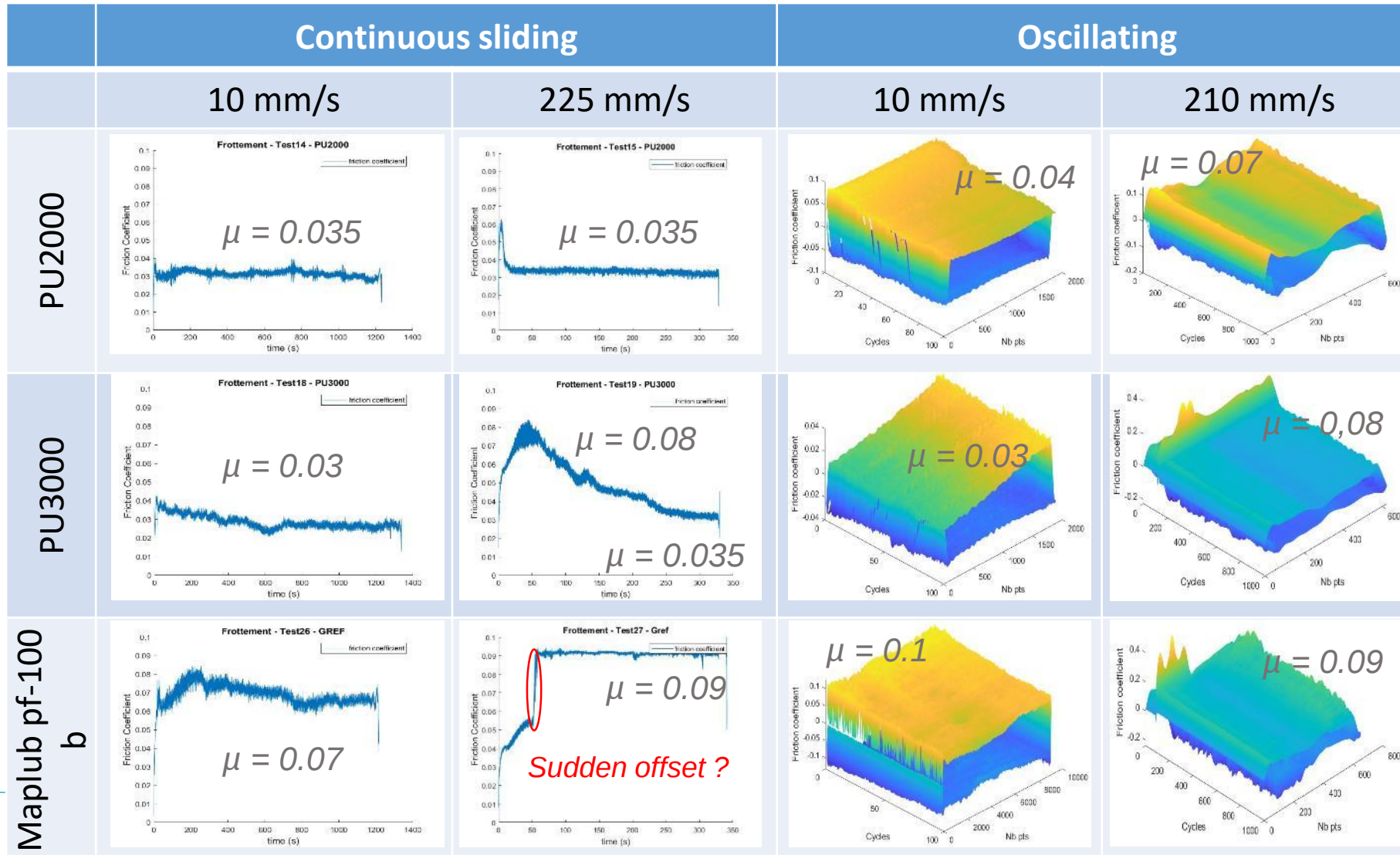
Sample cleaning

Topography and surface morphologies



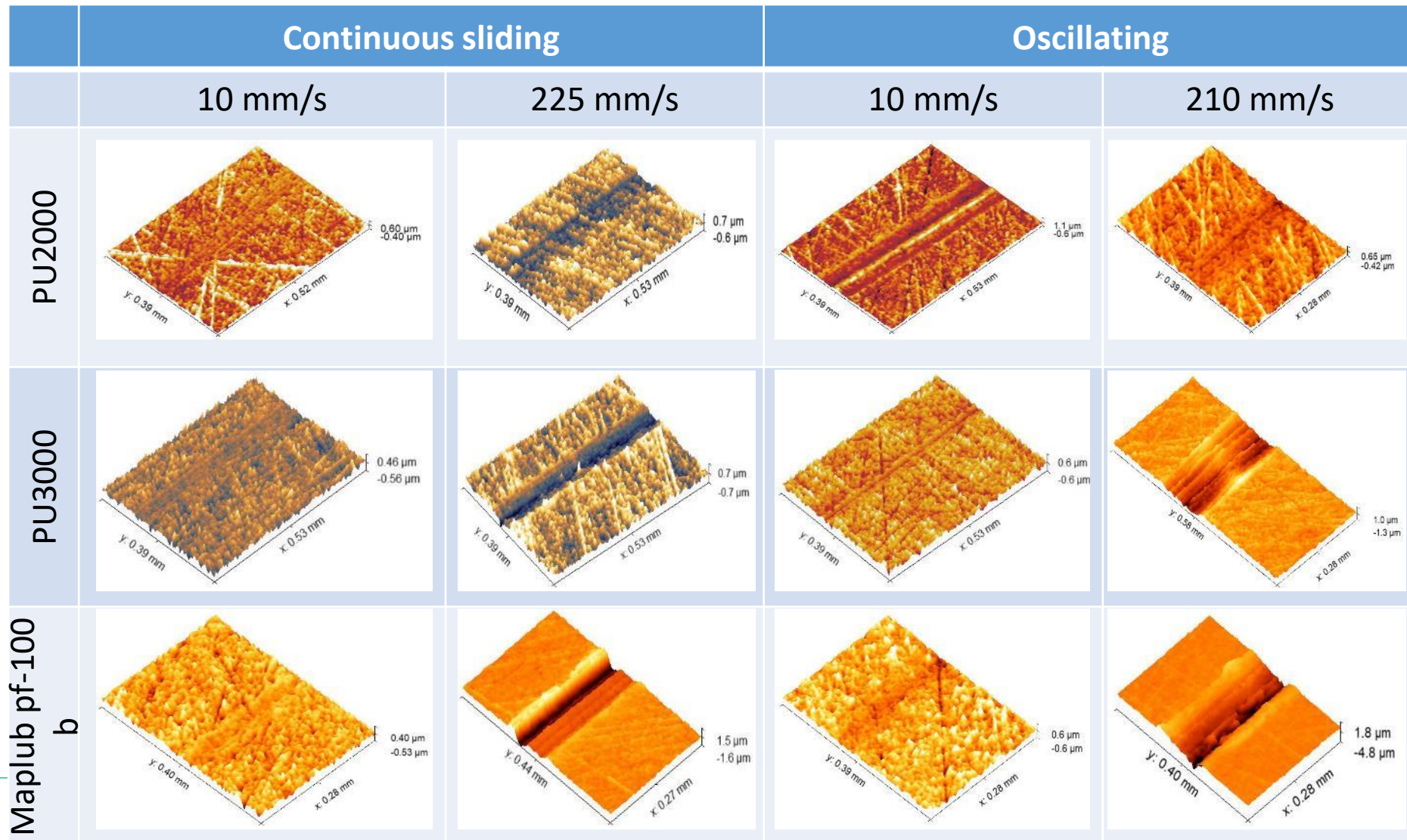
TRIBOLOGICAL TESTS ON HV TRIBOMETER - RESULTS

Pure sliding tests



TRIBOLOGICAL TESTS ON HV TRIBOMETER - RESULTS

Surface topography

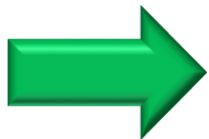
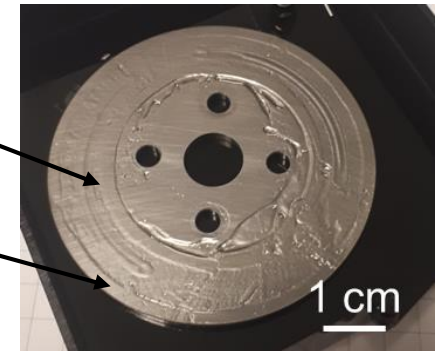


■ Conclusion

- ✓ **Two gelled hydrocarbon oils** (Multi-Alkyl Cyclopentane) **are obtained** (PU2000, medium Mn ; PU3000 high Mn)
- ✓ **Shear thinning behavior** favorable to dynamic bleeding
- ✓ Comfortable operating temperature range at first approach ($-40^{\circ}\text{C} \rightarrow +140^{\circ}\text{C}$)
- ✓ Stable behavior with **low torque in ball bearing** operation with no friction peak compared to classic space grease
- ✓ Tribological tests allowed to discriminate gelled oils and the reference grease :
 - PU2000 > PU3000 >> Maplub pf100-b
 - **Very good antiwear behavior** and better refill of the contact with PU2000
 - No lateral ejection of gelled oil on the disc

Friction
track

Disc remains
free of
lubricant



✓ **Hybrid technology as an alternative to classic space greases**

■ Continuation of activities

- ✓ ORO Test on rheometer to confirm good thixotropic behavior
- ✓ Vacuum endurance in bearings (RAMI bearing box) to study **stability and durability**
 - InS tribometer, linear and rotary motor with multi-couplings possible, 10^{-7} torr, max. 200N, max. 4,6 N/m, max. 1800 rpm, [-150°C ; +400°C]
- ✓ Rolling/sliding tests with SRR 10% @ FEMTO-ST
- ✓ Industrialization
 - Pilot-scale validation
 - Technology transfer

