INTERACTION BETWEEN BARRIER FILMS AND FLUID LUBRICANTS

Marchetti M.[*], Meurisse M.-H.[*], Vergne P.[*], Sicre J.[#], Durand M[+]. & Durand J.[§]

[*]LMC UMR CNRS/INSA 5514, bât. 113, 20 avenue Einstein, 69621 Villeurbanne cedex, France
telephone : 00 33 4 72 43 89 38 / fax 00 33 4 78 89 09 80
E-mail : Mario.Marchetti@lmc.insa-lyon.fr

[#]CNES Toulouse, 18 avenue Edouard Belin, 31054 Toulouse cedex, France

[+]ALCATEL Cannes, 100 boulevard du Midi, BP 99, 06322 Cannes La Bocca, France

[§]LMPM, ENSCM, UMR 5635, 8 rue de l'Ecole Normale, 34296 Montpellier cedex 5, France

ABSTRACT

Experiments were conducted to evaluate both static and dynamic interactions between anti migration films and fluid lubricants. Two distinct barrier films were evaluated; a fluorinated polymethacrylate resin (PAMA) and a plasma C.V.D. made PTFE coating (Pl-PTFE). Oils selected are usual ones for space applications, respectively a Z-perfluoropolyalkylether (Z-PFPE) and a multiply alkylated cyclopentane (MAC).

Z-PFPE was almost inert with respect to both coatings. MAC showed a slight interaction with both barrier films, particularly with PAMA.

MAC colour changes under UV light. Its non-wetting ability decreases. The coating's cohesion is probably modified. The infrared signature of oil contains peaks of the coating and its fluorine content increases. Former results with similar products were encountered in literature. We could therefore assume that the MAC renders PAMA light molecular weight fractions soluble. Oil viscosity of both lubricants remains constant.

I- INTRODUCTION

An increasing number of space mechanisms (ball bearings, gyroscopes), operating in special conditions (ultra high vacuum, reduced gravity...), are lubricated with specific fluids [1, 2]. These oils spread spontaneously over surfaces on which they are deposited. If it is an advantage on critical zones (balls and races in ball bearings), it can also constitute a disadvantage. This free migration can cause an impoverishment in lubricant of tribological areas and be the source of irreversible damage. This phenomenon can be combined with other sources of lubricant loss (gravity, evaporation...). The lubricant can go on optical devices or be trapped in the clearances of mechanisms and so becomes useless. Observations of on board fluid lubricated systems always show uncontrolled oil spreading [3, 4]. That is why solid lubrication is often preferred to the fluid one [4-9]. Only few articles deal with anti-migration coatings in spatial technology [5, 10-19].

Anti migration films are generally fluorinated or silane polymers with very low surface tension (less than 20 mN.m^{-1}). Their role is to maintain the oil in a definite functional area, such as ball races in ball bearings. The interesting property in selecting such a polymer is its non-wetting power. A surface coated with a barrier film can not easily be wetted by most liquids. Nevertheless, some chemical interactions between lubricants and barrier films were already raised in the past. But this aspect was not deeply studied [1, 12, 15, 18, 24, 25]. We decided to investigate the possible interaction between non-wetting coatings and oils. The behaviour of different lubricants versus barrier films under static and dynamic solicitations, at different temperatures and for two different surface roughnesses was experimentally evaluated.

Static interactions were quantified by keeping immersed in oil 440C coated stainless steel coated. Dynamic interaction was tested by imposing a rotational flow of oil onto the coated surface. Two distinct barrier films were evaluated; a classical one based on a fluorinated polyalkylmethacrylate resin (PAMA) and a plasma C.V.D. made PTFE coating (Pl-PTFE). The two oils selected are usual ones for space applications, respectively a Z-perfluoropolyalkylether (PFPE) and a multiply alkylated cyclopentane (MAC).

II- NON WETTING FILMS AND SPACE MECHANISMS

The wetting phenomena involve solid, liquid and gaseous phases of different materials [20]. The surface tensions γ_{LV}, γ_{SL}, γ_{SV} are the energies required to increase by a unit area respectively the liquid-gas, solid-liquid and solid-gas interface. The equilibrium of a liquid drop on a smooth surface is given by the Young-Dupré equation $\gamma_{LV}.\cos(\theta)=\gamma_{SV} - \gamma_{SL}$, where θ is the static wetting angle. A schematic representation, where γ_{LV}, γ_{SL}, γ_{SV} are interpreted as vectorial quantities is given in Figure 1.

According to Zisman theory [20], a fluid the γ_{LV} of which is lower than the γ_c of the substrate on which it is deposited will completely wet it. For a given material, γ_c is obtained by extrapolating the γ_{LV} values obtained for different liquids of a same family to the 0° wetting angle. A classification of γ_c values of chemical radicals, proposed by Zisman, is presented in Table 1. Surface roughness, fluid movement, chemical and physical surface heterogeneities make the phenomenon much more complex. Wettability can be evaluated by measuring the wetting angle of liquid drops deposited on a surface (see Figure 1).

Used since the sixties [10-12, 21, 22], non-wettable polymers are mainly based on fluorinated polyalkylmethacrylate (PAMA), containing fluorinated groups CF_x (x=1, 2 or 3). The efficiency of such polymers is evaluated thanks to five criteria: adhesion to the substrate [16], continuity, homogeneity, thickness of the coating and the non wettable ability. The first three parameters are not controlled in the case of space mechanisms. Thickness varies from 5.10^{-2} µm to 3 mm [12, 15]. According to Kinzig *et al.* [15], thin films tend to reduce risks of scratching in case of shearing. Thickness does not influence wettability, which is essentially a function of the chemical structure of the polymer as shown in Figure 2. Non-wetting ability depends on the concentration of fluorinated groups and on the organization of polymer chains in the coating structure [12, 15, 22, 23]. Measurements are also roughness and contamination highly sensitive. The possible interaction between a lubricant and a barrier film were quantified by measuring the surface tension of the barrier film.

III- MATERIALS

Two different polymers were considered in this work, a fluorinated polyalkylmethacrylate (PAMA), dissolved in n-butyl acetate, fluorescent under UV irradiation (λ=365 nm), and a plasma CVD made polytetrafluoroethylene coating (Pl-PTFE). Fluorescence is due to the presence of fluorinated organic molecules soluble in the polymer solution. Note that the two oils are not fluorescent under UV irradiation. This property was employed to detect the presence of the coating on the substrate and in the oils.

Both polymers were deposited on 440C stainless steel substrates where roughness is well controlled (Ra=1 or 0,15 µm). PAMA is applied thanks to a dip coating method on 80x20 mm^2 samples and on 70 mm diameter discs. Film thickness is around 20 µm. It was measured by weighting the samples after having dried them at 110°C. Pl-PTFE coating is obtained on 40x20 mm^2 substrates in a radiofrequency plasma reactor. Thickness, determined by ellipsometry, is between 490 and 730 nm. Before coating, all steel samples were cleaned in an ultrasonic bath at 60°C using an alkaline soap in water, then in ethanol and finally in acetone.

IV- EXPERIMENTAL PROCEDURES

IV.1- Static experiments

The objective was to determine if any interaction occurs when a 440C substrate of given roughness coated with a non wetting polymer is being immersed during a long period (at least 10 days) in a lubricant. Operating conditions are room temperature and ultra high vacuum. The area of interface has been chosen greater than the actual ones in space mechanisms in order to magnify an eventual interaction.

At the end of the test, samples of lubricant near the coated substrate, in the bulk fluid top and at the bottom of container were collected and analyzed using infrared spectroscopy. Lubricants and coatings were observed under UV light (λ=365 nm) in the case of PAMA. In case of suspected interaction between PAMA and oil, the lubricant fluorine amount is measured and compared to its initial value. The wettability of each coating was studied, after having blown the samples with dry and clean air to remove oil residues, by measuring contact angles of drops of distilled water and diiodomethane: the surface tension γ_c of the substrate was deduced from these values.

IV.2- Dynamic experiments

Convective or shearing effects in the oil could enhance static interaction. So, a rotational flow was created by using a rheometer. Such a device allowed speed (0,4 to 2 m.s^{-1}) and temperature (20 and 55°C) controls. In the cone - plate configuration, we could measure an eventual evolution of viscosity, which could be a consequence of the modification of the oil content. A scheme of the experiment is presented in Figure 3. Only PAMA was considered. After each test, oil was collected, submitted to UV radiation and analyzed by FTIR. The amount of fluorine was only determined in the case of a transfer of fluorescence from the coating to the lubricant.

V- RESULTS

V.1- Static interaction

PFPE is completely inert with both coatings. No oil fluorescence was observed after a 10-day immersion in the case of PAMA. The infrared signature of the lubricant was unchanged after contact with PAMA and PTFE. The wetting angle of liquid drops and surface tension of the coating were the same in the case of zones which had or had not been immersed, as shown in Table 2 (part a) (γ_{LV}=21,1 mN.m^{-1} at 20°C for Z-PFPE). We obtained the same conclusions for the Pl-PTFE. For this reason, we did not proceed any further analysis. Wetting angles and surface tensions values are presented in Table 2 (part b).

On the other hand, MAC interacts with both coatings. A 10-day immersion of PAMA causes a fluorescence of the lubricant. The zone in contact with oil appears clearly, with a deterioration of its fluorescence, as shown in Figure 4. Enlargements of infrared spectra in the 1730 cm^{-1} zone, corresponding to the methacrylate vibration group, indicate a weak presence of this chemical radical in oil after the test while not present in pure MAC. This transfer of matter from the coating to the lubricant is confirmed through the determination of fluorine content. Table 3 (part a) summarizes the analysis made on the pure product and on the one after the test. A surface tension's increase of immersed PAMA indicates a material easier to wet (Table 4 (part a)), which again confirms an interaction.

Pl-PTFE does not seem to interact with MAC. The infrared signature of the lubricant remains unchanged. However the γ_c deduced from the immersed Pl-PTFE and collected in Table 4 (part b) is close to the one we could measure on a MAC layer (γ_{LV}=30,7 mN.m^{-1} at 20°C). Because the coating is very thin, peaks and valleys of the initial substrate surface is remained: that is why oil is consequently trapped into the surface defaults. Nevertheless, the determination of fluorine content (Table 3 (part b)) shows here again a transfer of matter from the Pl-PTFE to MAC oil, indicating that the coating is not stable with this fluid. Despite this weak transfer, it is not possible to conclude to the occurrence of a clear interaction between Pl-PTFE and MAC. Moreover, we observed that capillary effects due to initial surface roughness were able to wet the "non wettable" coating.

In order to investigate whether time influences the rate of interaction between PAMA and MAC, a 40-day immersion test was conducted. Substrates of different roughnesses (Ra=0,04, 0,15 and 1 µm) were coated with PAMA and immersed in the lubricant. Operating conditions were the same as before. Observation under UV light showed fluorescence of the oil and a decrease in the coatings fluorescence in all cases, as previously shown in Figure 4. Wetting angles and fluorine amounts in lubricant are respectively given in Tables 4 (part c) and 3 (part c). Results are not quite coherent. MAC became fluorescent in all cases. But the small decrease in its non wetting ability did not correspond to an increase in the fluorine amount in the case of 1 µm roughness. The wettability coated substrate with Ra=0,06 µm is unchanged while fluorine amount in the collected oil increases.

V.2- Dynamic behaviour

As in static tests, PFPE is totally inert with PAMA, at 20°C and at 55°C. Both aspects of oil and coating remained unchanged under UV light.

MAC and PAMA showed the same tendency as during static experiments. A 5-hour rotational flow (920 $\leq \dot{\gamma} \leq$ 4500 s^{-1}) at 20°C did not deteriorate the fluorescence of the polymer, as can be seen in Figures 5a and 5b. But the lubricant became fluorescent compared to MAC before test (Figures 5c, 5d, 5e). The 5-hour experiments conducted at 55°C showed a well marked change in the coating response to UV irradiation (in the zone submitted to shear, Figures 6a and 6b) and an important fluorescence of the lubricant, presented in Figures 6c and 6d. The oil fluorescence was already observed after a one-hour test. But the infrared signature of oil samples collected after the tests was exactly the one of pure liquid, even in the 1730 cm^{-1} region. The determination of fluorine concentration in these samples did not show strong deviations compared to pure MAC. Although oil content was modified during the tests, no viscosity change of both lubricants has been detected.

Rotational flow and temperature have enhanced the interaction between MAC and PAMA, reducing the experimental time from several days to a couple of hours. Nevertheless, oil composition seemed unchanged. No preferential wrenching of the coating was observed according to substrate roughness.

VI- DISCUSSION

Static and dynamic interactions between PAMA coating and MAC oil lead to a modification of the polymer fluorescence under UV radiation and a small decrease in its non-wetting ability. The lubricant became fluorescent, its infrared signature revealed a weak presence of the PAMA radical and its fluorine content showed a small increase.

These observations could be caused by three different phenomena: dissolution of PAMA by the oil, partial solubility of small molecular weights fractions of the polymer, or diffusion of the UV sensitive additive from the coating to the liquid phase.

1) Because of the chemical structures of the products, the first assumption should be eliminated: rupture of bonds in PAMA molecules by MAC is almost impossible. PAMA is the result of polymerization of double carbon-carbon bonds to form covalent ones. These chemical bonds are too stable to be broken in the presence of MAC.

2) Assumption concerning partial solubility is based on former studies on PAMA interaction with fluids that are not solvent of this polymer [1, 12, 15, 18, 22, 24-26]. All these polymers are indeed constituted of molecules distributed over large ranges. Cohesion of these polymeric chains is in particular due to hydrogen bonds, as shown in Figure 7. The shorter these chains are, the weaker is the cohesion. The large amount of hydrogen atoms brought by each molecules of MAC (more than one hundred) associated with a temperature increase could cause a cooperative effect leading to a damage of the coating cohesion, as suggested in Figure 7. So, the molecules responsible for the fluorescence, initially trapped in the bulk and at the surface, were progressively released in the lubricant in contact with the substrate. The large specific area due to asperities,

revealed by an Atomic Force Microscopy analysis of PAMA tends to make the interaction easier. Fluorescence of MAC was always observed after interaction with PAMA. We assume that each PAMA part loss by the barrier film brings an amount of UV sensitive tracer in the oil. Moreover, a very small quantity of UV sensitive molecules is easily detected under UV light thanks to the high radiation intensity.

We can so explain the weak change in the infrared spectrum of MAC oil and the small variation in fluorine concentration after static and dynamic experiments in spite of an important fluorescence. The loss of the low molecular weight fractions causing a decrease in CF_x radicals density can explain the change in the surface tension of PAMA. We are therefore in presence of a fluid structure closer to that of n-butyl acetate, the solvent of PAMA, than to that of PFPE. Hydrogen atoms from MAC tend to create bonds close to these existing in a solution of PAMA in n-butyl acetate.

Prat **[1]** met the same problem with another polyalkylmethacrylate (solved in a fluorinated solvent) and a Z-PFPE. The barrier film failed to confine the fluorinated oil, which tended to progressively wet it, while in his case, the MAC completely remained in the functional area. Interaction between the Z-PFPE and this polyalkylmethacrylate is similar to the one of the coating with its fluorinated solvent.

In the case of the Pl-PTFE, the increase in fluorine content (Table 3, part b) can be attributed to the lack of cohesion of this polymer. This property could be improved by modifying preparation conditions.

3) The last interaction considered could be diffusion of UV sensitive molecules from the PAMA towards the oil. Low lubricant viscosity, solubility of the product in the fluid and temperature could enhance the phenomenon. Nevertheless, fluorescence was never observed with the PFPE, which structure is more compatible with fluorinated compounds, even at 55°C where PFPE and MAC viscosities are close together (respectively 0,18 and 0,24 Pa.s). This shows that diffusion, which is a process mainly controlled by the "solvent" viscosity, has not occurred during our tests.

VII- CONCLUSION

This study has shown the stability of PAMA coating with the PFPE lubricant: no deterioration of its color, no change in the surface energy of the coating and no modification in the infrared signature of the oil.

MAC interacts with PAMA. The polymer UV fluorescence is transmitted to the liquid. Temperature enhances this effect. The fluorine concentration in the lubricant lightly increases. Nevertheless, a weak signal of methacrylate radical is present in the infrared spectrum of the oil after test. The surface energy of the coated zone in contact with the lubricant increases, indicating a surface easier to wet. The coating on surface with the largest roughness seems to be the most

affected. According to the structures of these two materials, we can assume that MAC renders low molecular weights fractions constituting the PAMA coating soluble because of a cooperative effect of hydrogen bonds carried by the lubricant.

The Pl-PTFE has an interesting low surface energy. It could be easily applied on complex shapes. But its cohesion has still to be improved.

A useful conclusion of this study is the incidence of such interaction on the running of lubricated mechanisms. If barrier films based on PAMA are not stable, could a 10-year mission be fulfilled? What will be the evolution of the lubricant, the kinetics of this interaction being unknown? A simple way to answer these questions could be to select a non wetting coating according to the chemical nature of the lubricant it must confine: a polymer solved in a conventional solvent in the case of PFPE oils, and a coating soluble in a fluorinated solvent for synthetic hydrocarbon fluids as MAC.

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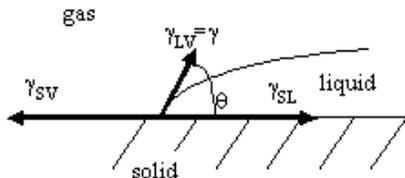27- Wicks Z.W. Jr et al, "Organic Coatings: Science and Technology. Vol. I", John Wiley and Sons, 1992.

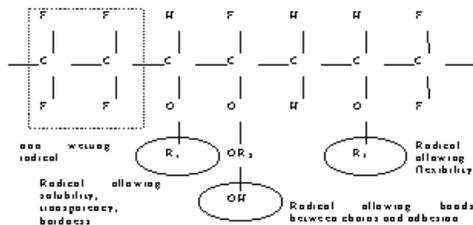*Figure 1 : Equilibrium of a liquid drop on a smooth surface*

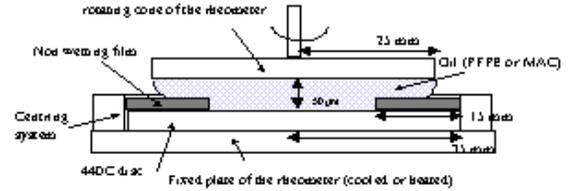*Figure 2 : General chemical structure of a non wetting polymer [27]*

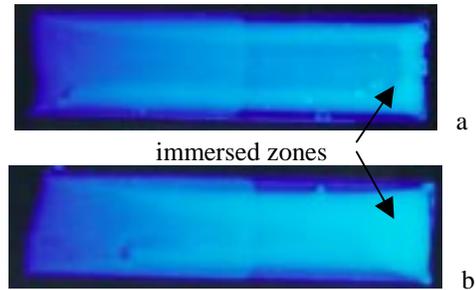*Figure 3 : Dynamic interaction between a non-wetting coating and a lubricant*

immersed zones

Figure 4: Aspect under UV light of 440C PAMA coated after a 10-day immersion in MAC
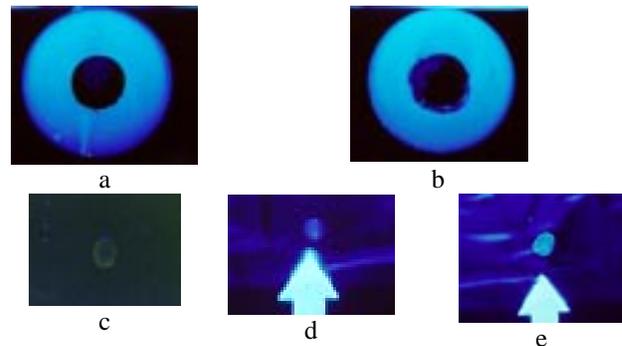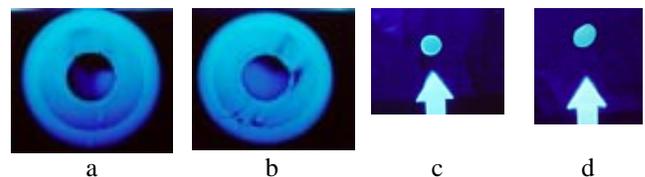*a) Ra=0,15 μm*
b) Ra= 1 μm

Figure 5: UV light visualisations after tests run at 20°C:
a) PAMA coating submitted to a rotational flow of MAC oil on substrate of Ra=0,15 μm
b) idem but Ra=1μm
c) a drop of pure MAC oil
d) a drop of MAC oil after rotational flow on coated substrate of Ra=0,15 μm
e) idem but Ra=1 μm

Figure 6: UV light visualisations after tests run at 55°C:
a) PAMA coating submitted to a rotational flow of MAC oil on substrate of Ra=0,15 μm
b) idem but Ra=1μm
c) a drop of MAC oil after rotational flow on coated substrate of Ra=0,15 μm
d) idem but Ra=1 μm

"H" bond between

rupture of cohesion between

UV sensistive
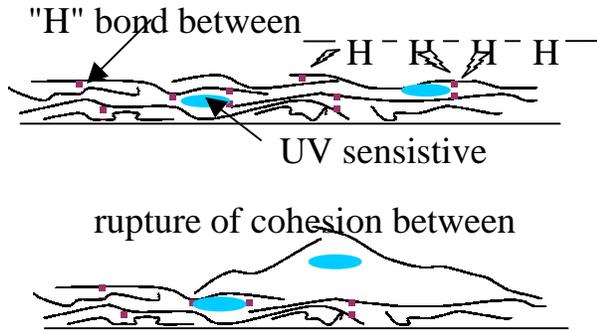
Figure 7: Cooperative effect deteriorating cohesion of PAMA coating

	radical	surface tension (mN.m^{-1})
	-CF$_3$	6
	-CF$_2$H	15
	-CF$_3$ et -CF$_2$-	17
	-CF$_2$-	18
	-²CH$_2$-CF$_3$-	20
	-CF$_2$-CFH-	22
	-CF$_2$-CH$_2$-	25
	-CFH-CH$_2$-	28
	-CH$_3$ (monolayer)	24
	-CH$_2$-	31
	-CH$_2$- et -CH-	33

Table 1: Surface tension of organic radicals [20]

	sample	Ra (µm)	Liquids H$_2$O	Liquids CH$_2$I$_2$	γ_c (mN.m^{-1})
a	PAMA (NI[1])	0,15	114,2	88,7	13,1
	PAMA (I[2])	0,15	114,5	88,5	13,1
	PAMA (NI)	1	115,0	91,0	12,0
	PAMA (I)	1	111,0	91,8	12,0
b	Pl-PTFE (NI)	0,15	103,9	82,6	16,4
	Pl-PTFE (I)	0,15	117,3	95,5	17,0
	Pl-PTFE (NI)	1	105,7	80,7	10,2
	Pl-PTFE (I)	1	118,5	93,7	10,9
c	440C steel (NI)	0,15	82,6	46,7	36,6
	440C steel (I)	0,15	96,8	39,0	39,7

[1] NI : non immersed in oil, [2] I : immersed in oil

Table 2 : Wetting angles (degrees) and surface tensions:
a) of PAMA 10-day immersed in PFPE and not plunged
b) of Pl-PTFE 10-day immersed in PFPE and not plunged
c) of 440C stainless steel alone immersed in PFPE and not plunged

			fluorine amount in MAC (ppm)		
			≤ 30 (pure MAC)		
	Sample	Ra (µm)	sample	surface	bottom
a	PAMA	0,15	**45**	≤ 30	≤ 30
		1	≤ 30	≤ 30	**147**
b	Pl-PTFE	0,15	≤ 30	≤ 30	**510**
		1	≤ 30	≤ 30	**59**
c	PAMA	0,06	**541**	≤ 30	≤ 30
		0,15	≤ 30	≤ 30	≤ 30
		1	≤ 30	≤ 30	≤ 30

Table 3: Evolution of fluorine amount in MAC at room temperature (according to the zone it was collected):
a) after 10-day static interaction with PAMA at room temperature,
b) after static interaction with Pl-PTFE,
c) after 40-day static interaction with PAMA

	Sample	Ra (µm)	Liquids H$_2$O	Liquids CH$_2$I$_2$	γ_c (mN.m^{-1})
a	PAMA (NI)	0,15	115,1	87,4	13,6
	PAMA (I)	0,15	112,1	86,2	14,2
	PAMA (NI)	1	115,4	88,9	12,9
	PAMA (I)	1	114,0	82,8	15,8
b	Pl-PTFE (NI)	0,15	103,9	82,6	16,4
	Pl-PTFE (I)	0,15	106,0	64,3	25,8
	Pl-PTFE (NI)	1	105,7	80,7	17,0
	Pl-PTFE (I)	1	107,2	57,4	30,3
c	PAMA (NI)	0,04	116,5	91,9	11,6
	PAMA (I)	0,04	112,	89,8	12,6
	PAMA (NI)	0,15	116,1	92,2	11,5
	PAMA (I)	0,15	113,7	93,9	11,0
	PAMA (NI)	1	117,1	91,4	11,9
	PAMA (I)	1	114,0	80,3	17,1

Table 4: Wetting angles (degrees) and surface tensions:
a) of PAMA (10-days immersed in MAC and not plunged),
b) of Pl-PTFE (10-day immersed in MAC and not plunged),
c) of PAMA (40-day immersion in MAC and not plunged).