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# New developments in PFPE lubrication: application to tinned electrical contacts

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**Abstract**— Electrical contacts performances and reliability can be improved by the use of a well-suited lubricant. Much work has been done and is still performed in this field. Different types of lubricants having different compositions and properties have been described. Perfluorinated polyethers (PFPE) are the lubricant class of choice when seeking high thermal and chemical stability, wide operating temperature ranges, and ultimately long lifetime and reliability. It has been shown in previous studies that linear and branched PFPE performed differently according to the contact surface finishes and that depending on the type of wear occurring in the contact different viscosities were beneficial. Indeed a universal lubricant improving the properties of all the various metallic finishes is still something to look forward to. In this paper, we show the first results on the friction and electrical properties of tinned electrical contacts lubricated with a new type of PFPE lubricant, Fomblin® HV25. This new PFPE oil has inherent high-viscosity, ca. 32000cSt (at 20°C), without any thickening agent (e.g. PTFE powder). Reciprocating friction tests were performed with a dedicated instrumented device. Improvements in friction reduction and in electrical behavior as compared PFPE oils with standard viscosities are described.

**Keywords**; tinned electrical contacts, friction, perfluorinated polyether lubricants, viscosity.

## I. INTRODUCTION

Early during the seventies it was shown that lubrication was an effective way to protect electrical contacts from the factors causing increases of contact resistance during their life time. This involves protecting the surfaces against high friction, wear and corrosion. A large review was published in chapter 6 of B. Chudnovsky's book [1] where all the lubricant types, liquids or greases are reviewed for electrical applications. In [2] a recent review of attempts to improve liquid lubricants is described. Improving lubricants involves adding nanoparticles, graphene, ionic liquids.

Our group has been working for many years on fluorinated liquids [3-5] (PFPE) because of their unique viscosity temperature characteristics, low vapor pressure and low surface tension. They have the advantage of being very inert. It was shown that they can remain on the surface of connectors after rinsing in solvent mixtures such as propanol and acetone [3] because they are not soluble in such solvents. They can wet most surfaces property which allows the formation of uniform films able to protect against oxidation and wear. It has been reported that greases for electrical connectors could show drawbacks such as dust contamination and removal during friction for example. Greases are composed of a liquid lubricant, a thickener and additives thus they are a more complicated “system” and describing the lubrication mechanisms of greases is very complex [6]. PFPE lubricants can have many different characteristics: linear or branched structures, a large range of viscosities and various functionalizations. Several works report studies on they what they call “gelled” oils with anti-creep capabilities [7, 8]. Recently Mainka et al. [9] have investigated the behavior of various PFPE with PTFE additives deposited on tin surfaces (1.9µm Sn on Cu base material). They varied the concentration of PTFE additive, the type and viscosity of various PFPE. Looking at the various values of the contact resistance  $R_c$  and the friction coefficients  $\mu$  of their work it can be concluded that lower  $\mu$  are obtained for low viscosity branched PFPE and for high viscosity linear ones. The very low friction coefficient measured for the linear PFPE led to quickly rising  $R_c$  values. During fretting tests all the  $\mu$  values were low; formulations with PTFE and linear PFPE of both viscosities (200-400cSt) displayed  $R_c$  values higher than 20mΩ by the end of the test. The formulation of PFPE and PTFE seems to have been optimized for branched PFPE.

In previous work [4] we have shown that linear PFPE performed better than branched ones for hot dipped tin contacts even after heating periods up to 500h at 150°C. Here we

present the first results on the behaviour of linear PFPE with high viscosities for the lubrication of tin electrical contacts.

It is interesting to study lubrication of tin coatings because the balance between lowering friction coefficient and maintaining low contact resistance is difficult due to the oxidation of tin.

## II. BACKGROUND AND RESULTS

### A. Samples

Previous studies were performed in our laboratory on the characterization of several PFPE lubricants. The effects of the structure of the lubricant (branched or linear) and of its characteristics were investigated during large amplitude friction tests and/or fretting ones. Several different coatings such as gold, silver, nickel were studied but most of the results were obtained with hot dipped tin on cuprous substrates. It was also shown that lubricants had different behaviors according to the nature of the metallic coatings because of the different wear mechanisms involved.

This chapter reports on large amplitude friction tests involving sphere on plane contacts made out of copper alloy with hot dipped tin coating (air knife wiped, 2 to 3 $\mu$ m thick) under a normal force of 2.5N (leading to a mean pressure of 700MPa).

### B. Friction device

Contacts were submitted to reciprocating wear cycles of 1mm at a constant speed of 0.5mm/s with a normal load of 2.5N. A dedicated device [4-5, 10-11] allowed recording the instantaneous values of the tangential force  $F_t$  during sliding. The mean friction coefficient per cycle  $\mu$  was then calculated from 2400 values of  $F_t$ . At each end of the wear track, the contact resistance was measured in static conditions (thanks to a feedback loop). A four-wire type set-up, involving a Keithley 181 nanovoltmeter, a Keithley 617 electrometer and a Keithley 228A V/C DC voltage/current source were used to measure the contact resistance. The mean value of 10 measurements of the voltage  $V$  for  $I=+10$ mA and  $I=-10$ mA was calculated to avoid thermal EMF effects (the open circuit voltage was set at a value of 200mV; this limitation was never active). Note that two values of  $R_c$  are measured per cycles, corresponding to each end of the wear track; there are thus two curves on the graphs showing the evolution of the contact resistance versus the number of cycles (straight line and dashed line).

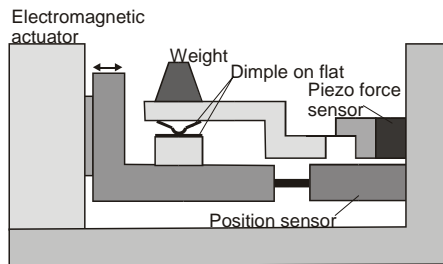


Figure 1. Friction device. A feedback loop controls the distance, speed and stops at the end of the wear track for the measurement of  $R_c$  in static.

### C. Lubricants

Before lubrication the samples were ultrasonically cleaned in acetone and propanol for 15min. Fomblin<sup>®</sup>YR1800 (YR1800) and Fomblin<sup>®</sup>Z25 (Z25), a branched and a linear PFPE (see table I), were deposited after dilution in Galden<sup>®</sup>SV90 (G90). Different dilutions were studied. After drying, lubrication of the flat coupons (2cmx2cm) was performed by dipping in the solutions for 15min and withdrawing at constant speed with a dedicated device. The speed was estimated to be 1cm/s. The process of dip coating is known to be reproducible way to deposit a thin film of liquid. This speed  $v$  is known to influence the thickness of the coating. For a Newtonian fluid, the thickness  $e$  of the coating is related to  $(v \nu)^{2/3}$  in the Landau Levitch equation [12] with  $v$  the withdrawal speed and  $\nu$  the viscosity of the solution.

TABLE I. PFPE STRUCTURE

PFPE	Chemical structure
Z25	$CF_3-O-[CF_2-CF_2-O]_m-(CF_2-O)_n-CF_3$
YR1800	$CF_3-O-[CF(CF_3)-CF_2-O]_m-(CF_2-O)_n-CF_3$

YR1800 has a kinematic viscosity  $\nu=1800$ cSt at 20°C while for Z25  $\nu=260$ cSt. The thickness of the films formed with the two lubricants was measured by a weighing method and found to be around 2 $\mu$ m for both solutions of YR1800 and Z25 at 20%vol. This is because the viscosities of the solutions at 20%vol of YR1800 and Z25 in G90 do not vary much leading to the same thickness of lubricant for a given type of substrate.

Fig.2 shows the mean contact resistance  $R_c$  values and the mean friction coefficient  $\mu$  per cycles for YR1800 and Z25 at 20% dilution in G90. Y1800 displays  $R_c$  values that increase as soon as friction starts;  $\mu$  starts at 0.3 and decreases to a plateau value at 0.1 till 120 cycles. After that large fluctuations of  $\mu$  are then recorded and correlated to fluctuations of  $R_c$  (observations of the wear track showed that the intermetallic and/or copper substrate was reached). For the Z25 lubricant the first  $R_c$  values are about 3m $\Omega$  and decrease very quickly during the first cycle as the number of asperities in contact increases. This is correlated to a very low initial  $\mu$  value ( $\mu=0.02$ ) which increases during a phase corresponding to the plastic deformation of the tin layer. It was observed that this occurred without adhesive wear as in dry contacts, but merely by displacement of the low hardness tin layer, thanks to the linear lubricant. After a running in period  $\mu$  reaches a very low value of 0.06. During the 200 cycles test, Fig.2 shows that  $R_c$  increases slowly up to a value of 20m $\Omega$ . Two slopes can be observed; between 10 and 100 cycles the rate of increase of  $R_c$  is very slow and between 100 and 200 cycles it is faster. Our hypothesis is that the composition of contact interface could change after a certain threshold of wear leading to an increase of contact resistance.

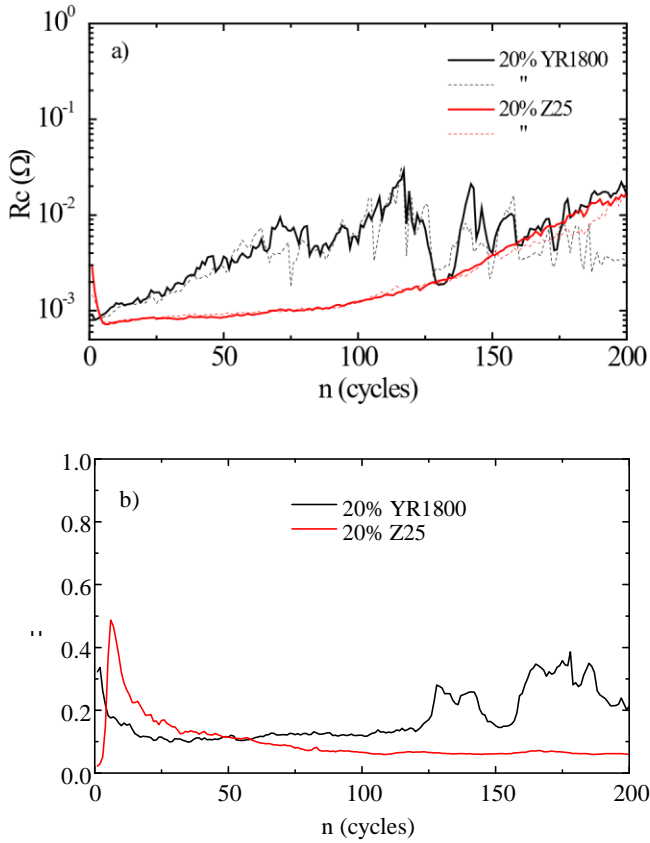


Figure 2. : Mean  $R_c$  value per cycle (a) at the end of the wear tracks and  $\mu$  values (b) during friction tests (1mm, 2.5N) of hot dipped tinned contacts lubricated with 20% YR1800 in G90 and 20% Z25 in G90.

Lubricated coupons were then heat treatment at 150°C for 500h (which is a relevant test for automotive connectors) in atmosphere: Fig. 3 shows that Z25 lubricant is characterized by constant and low values of  $R_c$  and  $\mu$  for 200 cycles while for YR1800 the lubricating effect stops at about 120 cycles. This behaviour was also observed during fretting tests performed on lubricated tin flats heat treated in the same conditions (not shown here). These results attested of a better lubrication effect with Z25 diluted at 20% in G90 for hot dipped tin contacts. This led us to favour this lubricant for further studies.

An optimization of the concentration of Z25 lubricant in Galden® SV90 was then performed. The results are shown in Fig.4 for concentrations of 3% vol, 10% vol and 20% vol in G90.

Very little lubrication effect is observed with solutions with a concentration of 3%. At this low concentration the lubricant is quickly eliminated from the interface and high friction occurs with  $\mu$  between 0.4 and 0.8. The  $R_c$  values are not very high because the copper substrate and/or tin-copper intermetallic layer is exposed and  $R_c$  corresponds to the copper/copper values. Measurement of the film thickness gave a value of  $e=0.17\mu\text{m}$  which is the order of magnitude of the roughness thus explaining the multiple metallic contacts and the severe wear of the surface. At 10% and 20% concentrations a strong lubricating effect is recorded. For the 10% solution the  $R_c$  evolution is about the same as with 20%; after 100 cycles  $R_c$  values increase slowly up to a value of 12mΩ. The initial  $\mu$  value is higher at the beginning for the 10% dilution but after

the running in period it is as low as for the 20% concentration solution ( $\mu=0.06$ ).

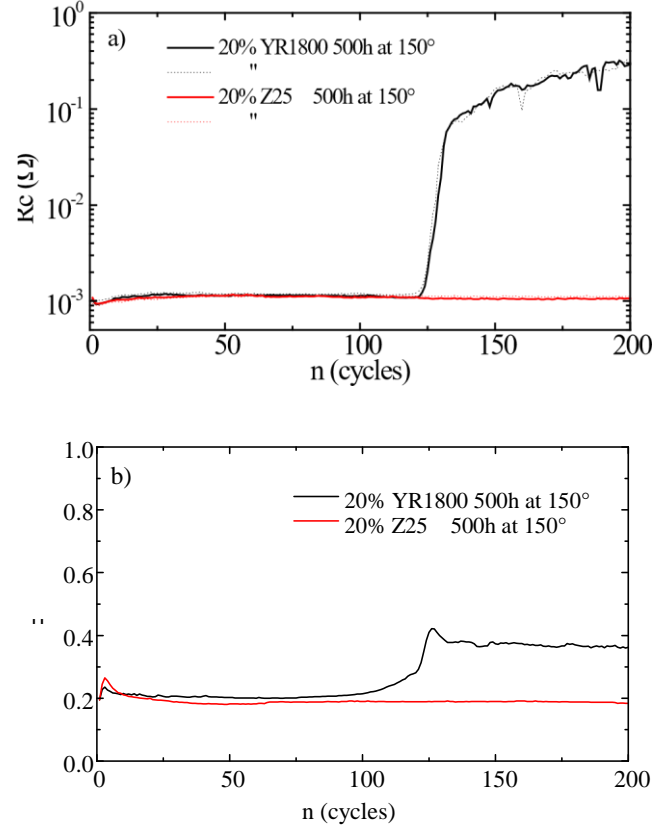


Figure 3. Same as Fig.1 . The lubricated flats were aged 500h at 150°C prior to the friction tests. A) mean  $R_c$  values and b) mean  $\mu$  values

These results showed that lubrication with Z25 at the 20% vol in G90 was a good solution to lower the friction force and the wear of tinned contacts with satisfactory electrical behavior for connectors in severe environment.

### III. NEW HIGH VISCOSITY LUBRICANT

A new type of linear lubricant Fomblin HV25® (HV25) was supplied by Solvay which allowed us to investigate the influence of the viscosity of a linear PFPE. The kinematic viscosity at 20°C is 32000cSt, its average molecular weight 55000amu and its evaporation weight loss after 22h at 20°C less than 0.3%.

The aim was to investigate friction behavior of such lubricants for applications where contact resistance values have to remain low. Note that Galden90®SV90 is not on the market anymore so that we had to use a different grade for this study.

#### A. Samples

The study was performed on rider on flat contacts of the same dimensions as above. They were made of copper alloy substrates electroplated with 1.6μm of nickel and 3μm of tin (this is different from the previous studies of II).

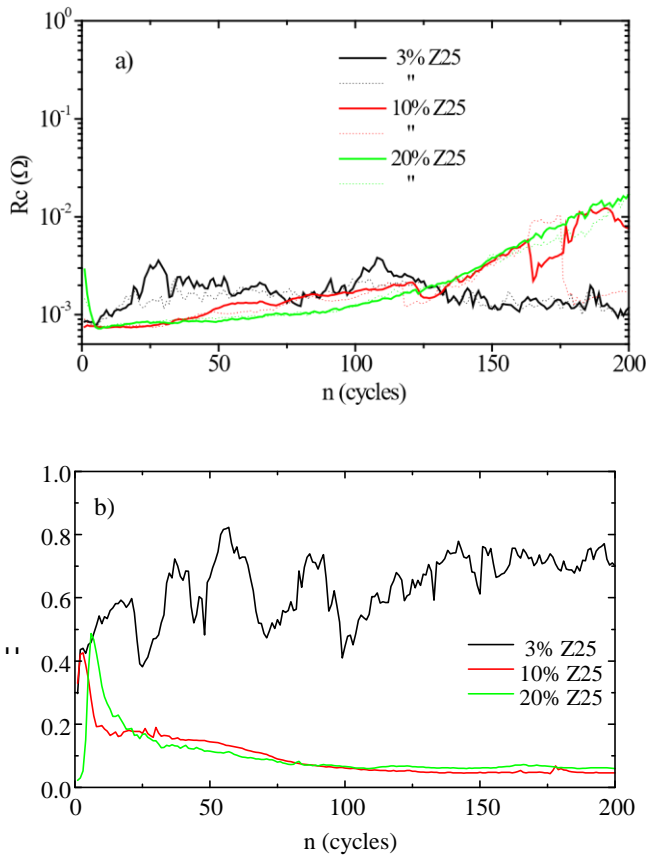


Figure 4. Mean  $R_c$  value per cycle (a) at the end of the wear tracks and  $\mu$  values (b) during friction tests (1mm, 2.5N) of hot dipped tinned contacts lubricated with Z25 at different concentrations in G90.

### B. Lubricant

The HV25 viscosity evolution with temperature is compared to that of Y type and Z lubricants in Fig. 5. The outstanding viscosity temperature behavior of Z lubricants can be observed.

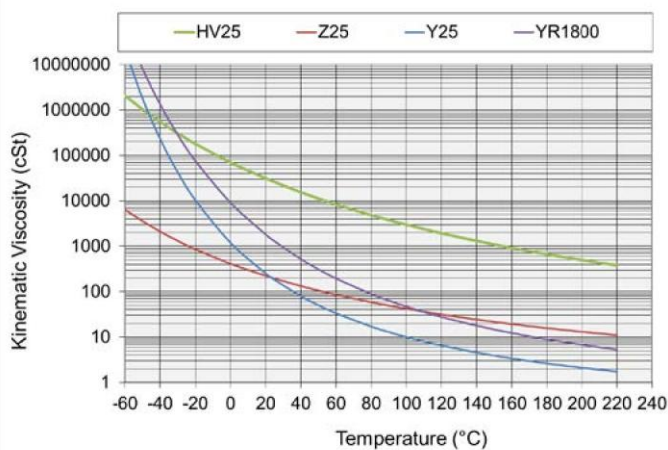


Figure 5. Variation of the kinematic viscosity of HV25 and of various linear and branched Fomblin<sup>®</sup>.

### C. Deposition process

Solutions of different concentrations (3% vol, 10% vol and 20% vol) were prepared by dilution of the lubricants in Galden<sup>®</sup>SV70 (G70). The flats were cleaned and then lubricated by dip coating in the same conditions as above.

After dip coating in the lubricant solutions samples were either dried in one step for 17h at room temperature or dried in a two steps process involving first a mild heat treatment of 45min at  $60^{\circ}\text{C}$  and then a 17h drying period at room temperature.

### D. Friction device

The friction conditions and the friction device were the same as in previous work reported. The influences of experimental conditions and of the friction device characteristics (feedback loops, stiffness) are very important in a comparative tribological study. Here they are the same which allows direct comparison. It is worth emphasizing that it is often difficult to compare data in literature because of the difference of experimental conditions, devices and measurement techniques.

### E. Characterization techniques

3D images were acquired with a Bruker Contour GT-X 3D Optical Profiler and surface roughness parameters were calculated with the device software.

A Thermo Scientific Phenom XL G2 Desktop SEM fitted with an EDX detector for imaging and elemental analysis was used.

## IV. RESULTS OF FRICTION TESTS WITH HV25 AND DISCUSSION

First the influence of the concentration of HV25 in Galden 70 was investigated. No example of lubrication for an electrical application with such viscous lubricants was found in the literature. In some works, thickening of the lubricant is achieved with thickening agents which is not the case here.

Fig. 6 shows the behaviour of films obtained by dip coating in solutions at 3%, 10% and 20% vol of HV25 in G70 and then dried in one step at room temperature for 17h. It can be seen on the friction coefficient curve Fig. 6 b) that the film at 3% is the first one to fail. After 60 cycles the film is eliminated from the surface,  $\mu$  increases, the tin layer is worn out and  $R_c$  decreases due to multiple copper/copper contact and oxidized debris. For 10% and 20% concentrations  $\mu$  remains extremely low ( $\mu=0.010-0.015$ ) for the 200 cycles.  $R_c$  values for solutions at 10% increase between 10 and 100m $\Omega$  while they remain under 20m $\Omega$  for the 20% concentration. Note that on Fig.5 b) the scale of  $\mu$  is from 0 to 0.2.

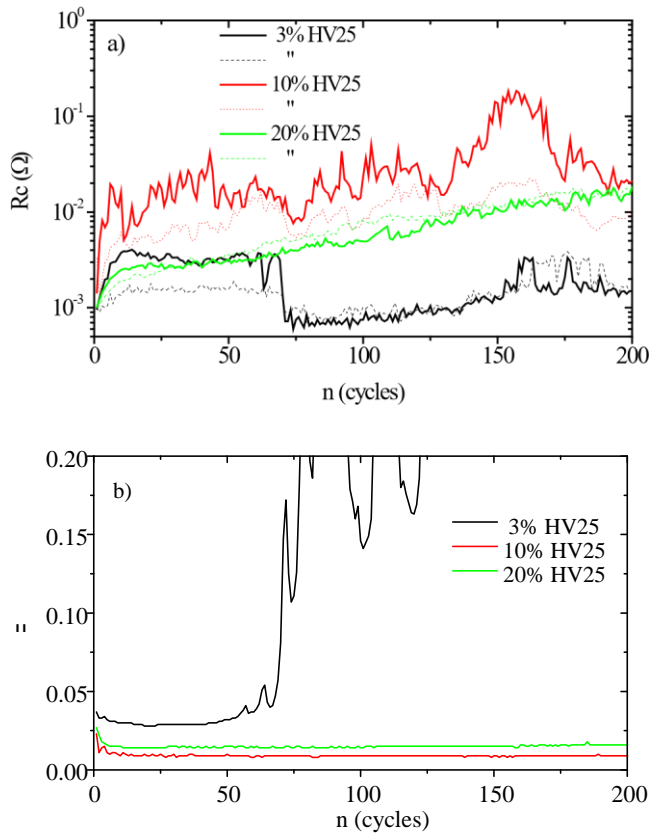


Figure 6. Mean  $R_c$  value per cycle (a) at the end of the wear tracks and  $\mu$  values (b) during friction tests (1mm, 2.5N) of hot dipped tinned contacts lubricated with HV25 at different concentrations in G70. One step drying. Note the change in scale for  $\mu$  in b).

The contact lubricated with HV25 at 3% displays a very low and rather constant  $\mu$  at 0.03 for about 60 cycles.  $R_c$  is about 3m $\Omega$ . After 60 cycles  $\mu$  increases very quickly while the contact resistance drops to 0.8m $\Omega$ . Nickel and copper were observed in the wear tracks, showing failure of the lubricating effect. For samples lubricated with the 10% and 20% solutions  $\mu$  remain very low ( $\mu=0.010$  to 0.015) for the 200 cycles.  $R_c$  values for 10% concentration varies from 10 to 100m $\Omega$ . For the 20% concentration  $R_c$  increases up to 15m $\Omega$ .

The roughness of these samples was measured to be 0.2 $\mu$ m. Two values control the contact resistance value : the mean value of the thickness of the film of HV25 and the thickness of the film at the asperities contacts. The former governs the friction coefficient and the latter ensures electrical conduction.

In [13] Homola et al. investigated the shear behavior of linear Z and branched Y Fomblin<sup>®</sup> fluids with a dedicated device and mica ultra smooth surfaces. They showed that when compressed by a very large force, the fluids between the mica surfaces could not be less than 7.4nm for the Y PFPE and 9nm for the Z PFPE.

They also showed that for very thin films of confined molecules a « surface viscosity » different from the bulk one had to be taken into account. Here the surfaces are rough and pressure on some of the asperities must allow a tunneling current to flow as shown by the low measured values of  $R_c$  (at the beginning of the test before eventual degradation).

The effect of a two-step drying was then investigated and showed an improvement of the friction and electrical properties for the three concentrations. In Fig. 7 (two-step drying) for the 3% concentration the lubricant elimination occurs after 180 cycles which is 3 times longer than in Fig. 6 (one-step drying). For the 10% and 20% films  $\mu$  remains constant and extremely low for 200 cycles. For the 10% film  $R_c$  values can be seen to vary between 1 and 20m $\Omega$  while for the 20% film HV25 film both very low  $\mu$  and very low  $R_c$  are recorded.

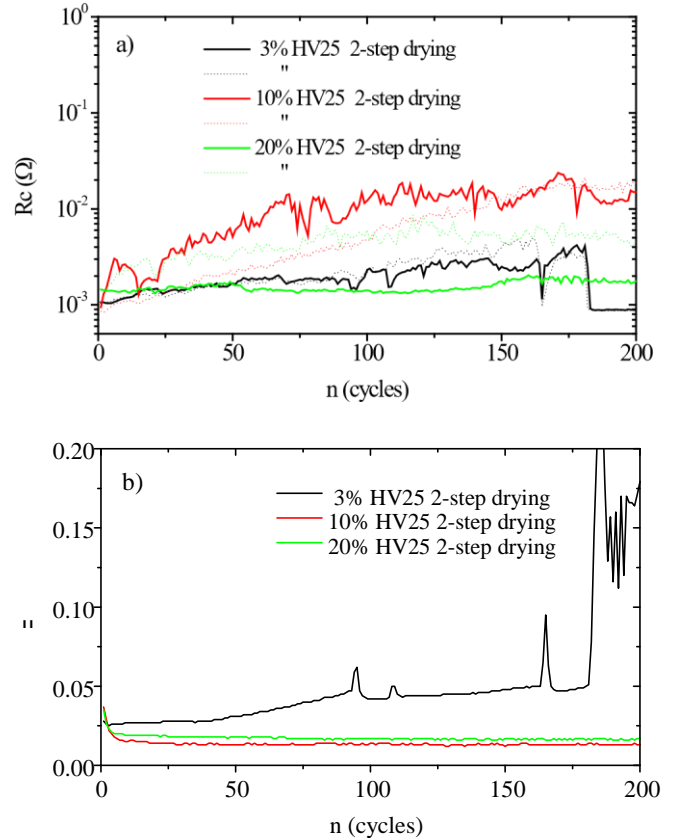


Figure 7. Mean  $R_c$  value per cycle (a) at the end of the wear tracks and  $\mu$  values (b) during friction tests (1mm, 2.5N) of hot dipped tinned contacts lubricated with HV25 at different concentrations in G70. Two steps drying.

Galden<sup>®</sup> SV70 is a low molecular weight branched PFPE with a boiling point of 70 $^{\circ}$ C. Drying the film 45min just below the boiling temperature allow the molecules to evaporate and leave an entanglement of flexible linear molecules displaying a more efficient tribological behavior.

New friction tests were then performed with Z25 diluted in Galden<sup>®</sup>SV70 in order to compare Z25 and HV25 with the same dilution in the same solvent and with the same coatings/substrates. The behaviors of the contacts lubricated with 20% vol HV25 and Z25 in G70 are represented in Fig. 8. Small but interesting differences in the  $R_c$  evolution with the number of cycles are shown in Fig. 8 a). Very large difference in friction coefficient  $\mu$  can be observed for these two different lubricants in Fig. 8 b).

HV25 provides a long lasting lubricating effect while Z25 shows a friction coefficient starting at a value of 0.26 then a peak at 0.3 and a decrease to 0.08 at 200cycles.  $R_c$  values for HV25 increase more than those of Z25; which is correlated to the very low friction values.

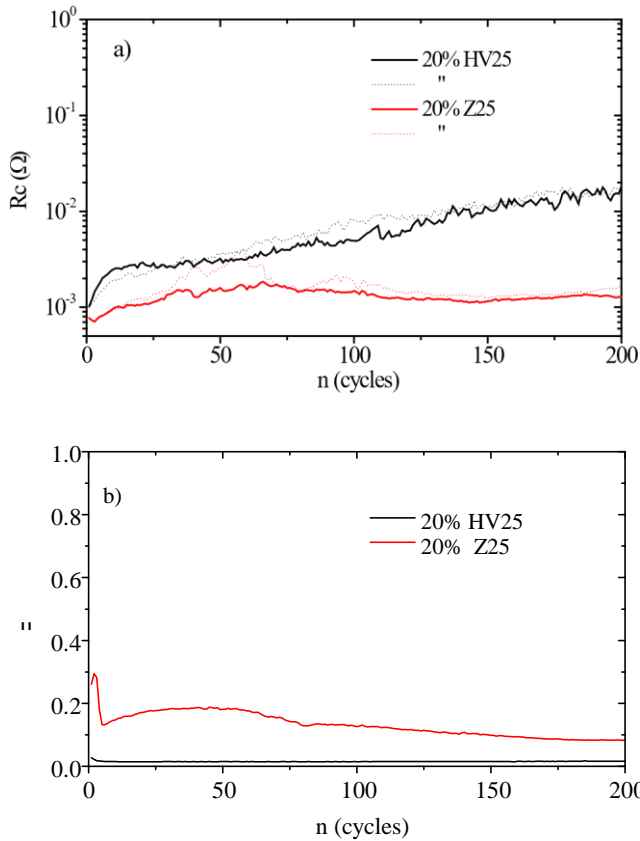


Figure 8. Mean  $R_c$  value per cycle (a) at the end of the wear tracks and  $\mu$  values (b) during friction tests (1mm, 2.5N) of tinned contacts lubricated with 20% HV25 in G70 and 20% Z25 in G70.

In order to better understand the friction reduction, the wear and the electrical behavior for these two lubricants, additional observations and characterizations of the wear tracks after 200 cycles were performed.

#### A. Observation of Z25 wear tracks

An optical profiler and a scanning electronic microscope were used to observe the wear tracks.

Fig. 9 represents the 3D profilometric view of the Z25 wear track on the flat after 200 cycles; it shows that during friction tin is removed from the surface and pushed towards the end of the wear track whose depth is about 2.2 $\mu$ m. The thickness of the tin layer on the nickel under coat was measured to be around 3 $\mu$ m; thus not all the tin has been removed but a large quantity. The observed heaps formed at the end of the wear track on the flat explain why the  $R_c$  values which are measured at the end of the wear track remain low (3m $\Omega$  after 200 cycles).

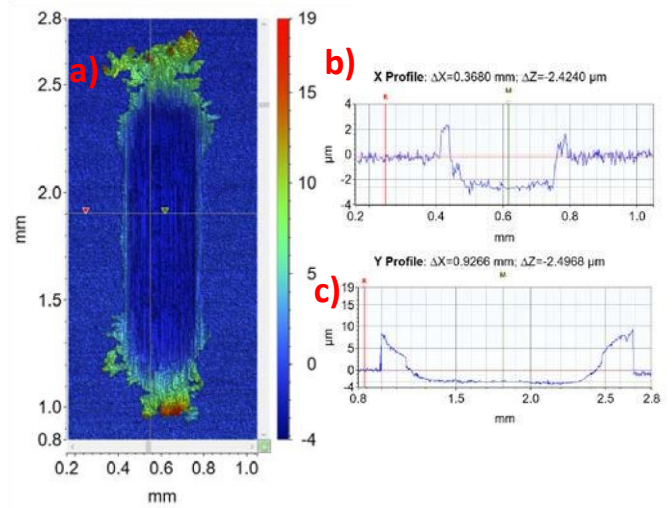


Figure 9. a) Profilometry 3D view of the 200 wear cycles with 20% Z25 G70 and extracted profiles b) and c) showing the depth of the wear track

SEM observations and EDX analysis were performed to obtain the composition.

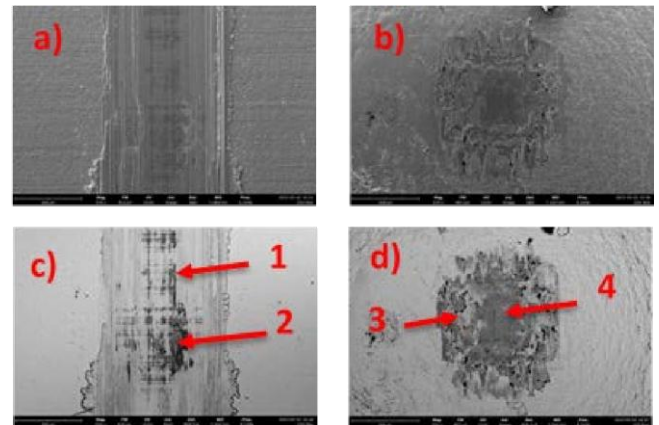


Figure 10. SEM images in SE mode (a and b) and in BSD mode (c and d) of the wear tracks lubricated with 20% Z25 in G70, showing the location of the EDX analysis. a) and c) flat, b) and d) rider

Fig. 10 a) and b) acquired in secondary electron (SE) mode show the rather smooth aspect of wear of the track on the flat (a) and the rider (b). On some locations of the images obtained in backscattered electron detector mode (BSD), (images c and d), differences in color are observed attesting that the composition is locally not homogeneous within the wear track. Brighter spots in the BSD images indicate the presence of high atomic number elements. Here tin has the highest atomic number while nickel and copper have lower ones. EDX analysis performed on the flat wear track in spot 1 (Fig.10 c) white aspect spot) indicate the presence of Ni and Sn with a ratio  $Ni/Sn$  of 0.04. This ratio in spot 2 (Fig. 10 c) dark spot) was 3.35 attesting of the detection of Ni in the depth of analysis which is about 1 $\mu$ m. In some locations the tin layer is thinner due to friction.

Fig. 10 b) and d) displaying the rider show an accumulation of compacted debris in the middle of the wear track on the rider. The  $Ni/Sn$  ratio obtained from the EDX analysis was 0.02 in spot 3 (white aspect) and 0.53 in spot 4 (dark aspect). On the side of the wear scar the white zones such as spot 3 contain

almost no Ni; this explains the low and constant value of  $R_c$  despite the severe wear.

### B. Observation of HV25 wear tracks

In the case of the contact lubricated with HV25 the wear situation is very different: Fig. 11 shows the 3D view of the wear track on the flat which is barely visible. The extracted profiles do not allow measuring any depth but only show a flattening of the asperities.

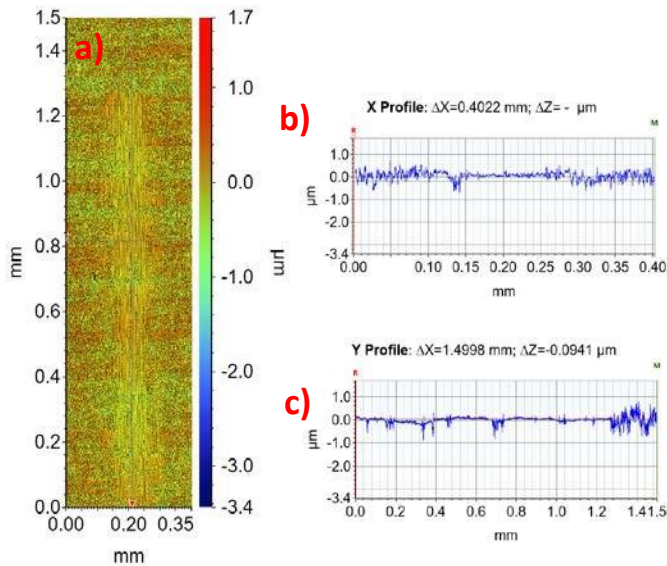


Figure 11. a) Profilometry 3D view of the 200 wear cycles with 20%HV25 and extracted profiles b) and c) showing the depth of the wear track

The roughness which was about  $0.2\mu\text{m}$  on the as deposited coupons was measured to be  $0.07\mu\text{m}$  inside the wear track. This is very consistent with the extremely low coefficient of friction

The SEM images of the wear scars on the ball and on the flat in SE mode are shown in Fig.12.

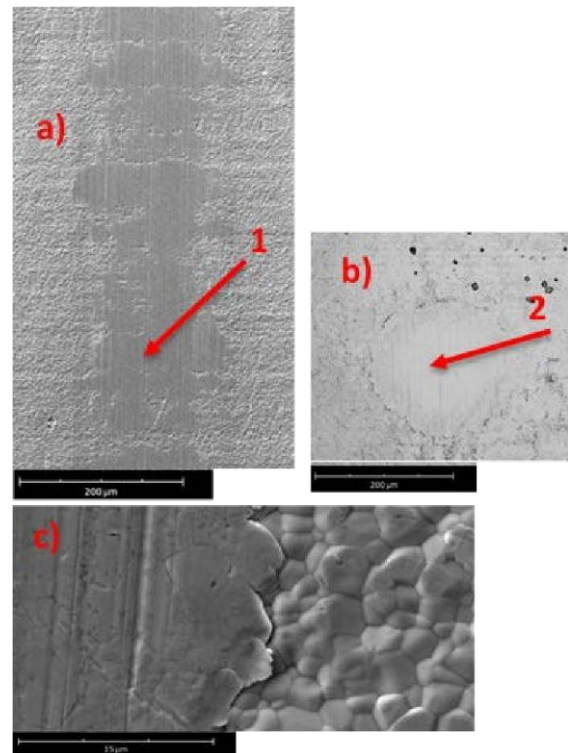


Figure 12. SEM image in SE mode of the wear tracks lubricated with 20% HV25 in G70 showing the location of the EDX analysis. a) flat, b) rider, c) zoom of the edge of the wear track of a). Full scale for images a) and b) is  $200\mu\text{m}$ ; full scale for image c) is  $15\mu\text{m}$

They attest of the high lubricating effect of the HV25 film. No plastic deformation of the tin layer has occurred. The lubricant film provided a cushioning effect increasing the load bearing area. The only effect seems to be the deformation of the top of the asperities with some smearing on the side of the rider and of the flat (Fig.12 c)). The EDX measurements for the flat wear track in spot 1 and the rider wear track in spot 2 did not display any nickel ( $Ni/Sn$  ratios equal to 0).

At this stage several points are worth investigating:

- are the differences in behavior with Z25 and HV25 solely due to a thickness effect? According to viscosity calculations for solutions, the 20%vol solution of Z25 in G70 has a viscosity of 1.82cSt while a 20%vol solution of HV25 in G70 has a viscosity of 5.32cSt. This could lead to a thicker layer during the dip coating deposition. But other characteristics such as the average molecular weight for example could have a role on the outstanding effect recorded for the HV25 film.

- how can the contact resistance values be so low? Current can flow either by direct metallic contact on small asperities or by tunnel effect at the asperities summits if the film can get thin enough ( $< \text{nm}$ ). Dedicated experiments on ultra-smooth surfaces could bring some insight on the question. When tin coating deformation occurs close contact of the spherical shape of the rider on the end of the wear track can lower the  $R_c$  values measured at the end of the wear tracks. Contact resistances in this work and previous ones are measured in static, at the end of the wear track thanks to the feedback loop. Although this is what corresponds best to a connector use, it is not always how contacts resistances are measured in literature, many authors measuring  $R_c$  during



friction. Here for HV25 20% in G70 the wear tracks on the flat do not show any groove so this effect does not account for the low values of Rc. Thus we suppose that these low values could be linked to the thinning of the lubricant under the normal load.

- what is the role of the solvent? We have observed during all our experiments with Z25 in G90 (some reported in paragraph II) that some of the Galden could stay trapped in the film and increase the lubricating effect ( $\mu$  initial=0.02). This effect was not recorded for 20%Z25 in G70 for which the initial friction coefficient was 0.3. This can be explained by the vapor pressure values of G70 and G90. Galden®SV70 has higher vapour pressure (141torr) than Galden®SV90 (between 62 and 17torr).

Therefore when characterizing the behavior of a lubricant much care must be taken in giving all the experimental conditions including the solvents dilutions and drying durations. This illustrates how difficult it is to compare data from the literature.

## V. CONCLUSION

Some results on the friction and electrical behaviour of branched and linear PFPE on tinned contacts have been summarized. They showed that linear lubricants applied to tin contacts had a better lubricating effect than the branched PFPE. New results on a recently synthesized linear PFPE with very high viscosity are shown. The friction coefficient values were extremely low (below 0.05) while the contact resistance remained between 2 and 20m $\Omega$  at the end of 200 friction cycles. From these results it can be inferred that for linear PFPE, a higher viscosity (HV25 as compared to Z25), can be beneficial in terms of friction and wear reduction with low values of the contact resistance at the same time. Under normal load the thickness of the linear PFPE can reduce to sub nanometric values allowing the asperities to be in quasi metallic contact thus allowing conduction. Because of the flexibility of the chain (and of the low surface tension) very efficient lubrication is ensured in the contact interface. Much more work is necessary to fully understand the results described here and to propose mechanisms of lubrication applying to high viscosity PFPE lubricants.

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