Friction and lifetime of a contact lubricated by a solid third body formed from an MoS_{1.6} coating at low temperature

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Abstract

A special test device has been designed to investigate the friction and wear behaviour of materials under very low temperature conditions. This paper describes an analysis of friction and wear in sliding contacts "lubricated" by a solid third body formed in situ from an MoS_{1.6} coating at 297 K, 223 K, 173 K and 123 K, under reciprocating kinematics in a gaseous atmosphere. It was found that under given test conditions, the friction coefficient was somewhat higher at low temperature than at room temperature under oxidising atmosphere. The lifetime was ten times lower at 123 K than at room temperature. Under no oxidising atmosphere the temperature does not effect on the friction value. The consequences of decreasing temperature on friction and lifetime are interpreted on the basis of experimental evidence and from the viewpoint of mechanics in order to evaluate third body flows and the rheology activated in the contact.

Keywords: Solid lubrication, Molybdenum sulphide, Third body, Low temperature, in situ tribometry

Nomenclature

μ	=	Friction coefficient
d	=	Displacement (μm)
R_a	=	Roughness (μm)
Q	=	Flow of third body
Q_s^i	=	Internal source flow
Q_i	=	Internal flow
Q_e	=	External flow
Q_r	=	Recirculation flow
Q_w	=	Wear flow
$*$	:	Symbol for the qualitative scale of flows
$+$	:	Symbol for the qualitative scale of cohesion
$\#$	:	Symbol for the qualitative scale of ductility
$S_{2,4}$	=	Screens
S_3	=	Bulk of third body
S_1, S_5	=	First bodies

1. Introduction

"Low temperature" covers an extensive range from 0 K to 273.15 K [1]. Temperatures under 120 K are known as "cryogenic", while those below 0.3 K are termed "super-low". Cryogenics technics are characterised both by low temperature and by special media: gaseous helium, liquid cryogen, vacuum. A bath of liquid cryogen is commonly used to cool the system: nitrogen (LN₂) at 77 K or helium (LHe) at 4.2 K. Various methods of cooling using gaseous and liquid environments have been implemented [2, 3].

In the 1990s, most research on friction and wear at "low temperatures" has been carried out in connection with space applications (telecommunications satellites, earth observations, scientific missions, etc.) [4-6] and laboratory "low temperature" tribosystems have been developed in several countries [1, 2, 7, 8, 9]. At higher temperature range 230K - 273.15 K the applications are related to inland transportation industries. At "low temperature", components with sliding surfaces in relative motion, such as bearings, seals or valves, are critical parts. Many space mechanisms use solid lubricants such as MoS₂, hard coatings (TiN, DLC), and combinations of materials with low friction and wear such as polymers vs steel. However, much remains to be understood on this subject as few in-depth studies have as yet been carried out on their tribological behavior in relation to temperature decrease. Few results have been published regarding "low temperature" [2, 3, 7-16].

Few studies focus on MoS_x solid lubricant [17-21]. Most interpretations of the friction values and lifetime at room temperature are related to parameters recorded before and after friction. They reveal that the friction coefficient is influenced by the structure and morphology of the coating, the applied load, the velocity speed of the first bodies, the environment, the layers adsorbed, etc. An extensive review [4] of the tribological properties of thin solid film lubricants, especially MoS₂ coatings, has been performed for spacecraft components and under specific spacecraft conditions. Nevertheless, the literature provides some information ~~and few interpretations~~ on the performances of MoS_x at low temperatures. The coatings can be solely MoS₂ [4, 16, 22, 23] but in most of the cases the study concerns polymer based solid lubricant coatings with MoS₂ pigments... In these types of coating the effect of temperature on wear resistance is ambiguous and depends mostly on the polymer binder (chemical nature, lubricant-to-binder weight ratio) [2, 9, 13, 15, 24]. ~~El Marazki [22] carried out test to characterize the frictional properties of~~

~~TIOLUBE 1175 which is an inorganic bonded dry film lubricant utilizing a specially formulated MoS₂ pigment working in combination with other lubricant pigments. Friction coefficient was determined between aluminium pads and aluminium coated with TIOLUBE 1175 at five normal loads under various environments. The average coefficients of friction increased with decreasing temperature at the same normal load.~~

~~Gamulya and all [14] have tested different MoS₂ coatings sprayed on steel substrate with a pin-on-disc tribometer under vacuum at room and low temperature. The coatings contained graphite and/or PTFE and/or compounds binders with various fraction. For all the coatings tested, with a decrease of the temperature from 293 K to 120 K there was a general prolongation of wear life and some increase of the friction coefficient. The fatigue mechanism of fracture of these coatings was realized in the case of a high degree of texturing of the lamellar lubricant component within the active layer, owing to plasticization of the binder during sliding.~~

~~It appears that very few data are available in the case of~~ Table 1 lists several sets of friction coefficient values obtained for solely sputtered MoS₂ coatings, at different low temperature under various environments. Under high vacuum or cryogenic liquid, the results from literature show that temperature has no effect on friction coefficient value.

Some results for solid lubrication in ball bearings under cryogenic conditions are reported in [4]. The ball bearings with films of sputtered MoS₂ were run at 18 K under vacuum. The bearings lubricated by sputtered MoS₂ enable their low torque performance during the test (further 2 million revolutions). The MoS₂ – coated bearings mounted with PTFE composite cages gave low torque in vacuum at room temperature but significantly highly torques at cryogenic temperature (x6) but still provided lubrication effect. Iwabuchi and al [11] examined frictional properties of a MoS₂ sputtered coating on a 316L steel specimen with a thickness of 1 µm. Reciprocating sliding was provided with a peak to peak amplitude of

100 μm . For a normal load of 10 N (i.e. an estimated maximal Hertzian pressure of 485 MPa) the friction coefficient was measured at 4 K in liquid helium.

Xu et al. investigated with a micro-tribometer reciprocating test machine the low temperature (down to 233 K) tribological behaviours of MoS₂ coating deposited on 1045 steel or on cast iron [16]. The application field was the automotive industry, especially to examine the potential application of solid-lubricating coatings on friction engine parts. At 233 K, compared to 273 K the wear volume was seven times higher. At 233 K the friction coefficient was kept at its initial level of 0.1 during 25000 seconds and then increased drastically to 0.6. Ball bearings [22] were lubricated with “ultra low friction” films and torque measurements were made at 20 K under vacuum. No significant changes in torque and therefore friction were observed on cooling from 300 K to 20 K. On the contrary, Karapetyan and Silin [23] showed that the friction coefficient with MoS₂, in vacuum, was almost constant from room temperature down to 240 K below which temperature it almost doubled reaching a new value at 190 K.

MoS ₂	T° (K)	Environment	Configuration or Contact pressure (MPa)	Friction coefficient
Sputtered [4]	18 293	Vacuum	Ball bearings	Low torque No T effect
MoS ₂ [9]	120 293			0.03 0.03
Sputtered [11]	4	Liquid He	485	~ 0.36
Ion plated Mo coating + ion sulphurisation process [16]	233	Oil	Cylinder/ring 2.67	0.1
Sputtered [22, 25]	300 to 20	Vacuum: 10 ⁻⁷ Pa	Ball bearings	Low torque No T effect
MoS ₂ [23]	77 120 170 220 293	Vacuum: 5.10 ⁻⁸ Torr (~6.10 ⁻⁶ Pa)		0.04 0.04 0.04 0.02 0.02

Table 1: Synthesis of studies on MoS₂ behaviour under low temperature

Based on experimental evidences, the objective of this paper is to investigate, for a given mechanism, how low temperature modifies the tribological behaviour of a contact lubricated via a MoS_{1.6} coating deposited in vapour phase by magnetron cathodic sputtering

in reactive mode [26, 27]. The temperatures for the tests presented in this paper are between 297 K and 123 K. This range of temperature is related to environmental conditions experienced by spacecraft mechanisms that are subjected to temperatures from cryogenic levels to several hundred Kelvin [4].

2. Experimental details and third body concepts

2.1. Experimental apparatus

The tests were carried out on the laboratory's simulator and are the subject of a detailed description in [17]. To investigate the effect of low temperature in a gaseous environment, a specific chamber was designed, shown in figure 1.

Before starting any tests, the chamber was first blasted with dry nitrogen gas at room temperature for 30 minutes, thus removing air from the chamber and preventing any condensate from forming on the sliding surfaces and in the test chamber. Then the dry gas was cooled before being injected into the test chamber allowing possible condensate in the cooling coil located before the test chamber. The gas is cooled by circulation in a cooling coil plunged into liquid nitrogen. The flow of the cooled dry gas was regulated and allowed cooling the chamber and the two specimens. The pressure in the test chamber was 1.52×10^5 Pa, higher than atmospheric pressure of 10^5 Pa, leading to an escape flow at the top of the chamber. The upper plane specimen was also cooled via its holder into which a cooling coil was inserted. The chamber and the specimens were cooled for one hour. Experiments can also be performed at room temperature in the same gaseous environment.

2.2. Materials and Operating conditions

The common parameters of the tests are:

- the lower specimen, a convex first body ($R_a = 0.1 \mu\text{m}$) made of AISI 304L steel,
- the upper specimen, a flat first body ($R_a = 0.1 \mu\text{m}$),

- reciprocating kinematics,
- the magnitude d of displacement of the upper first body is $\pm 50 \mu\text{m}$ (d is represented by arrows on the figures), the lower first body is fixed,
- a normal load of 200 N,
- ~~- maximal Hertzian pressure of 1 GPa.~~

Both the normal load and the resulting friction longitudinal force were continuously measured. The friction coefficient is defined as the ratio of the normal load and the longitudinal force.

“Blind” tests and visualisation tests were carried out in view to reconstituting the contact dynamics. For the “blind” tests the lower specimen was a non-coated AISI304L steel first body and the upper specimen was a coated AISI304L steel first body with a $1 \mu\text{m}$ thick $\text{MoS}_{1.6}$ coating. The $\text{MoS}_{1.6}$ coating was deposited in vapour phase by magnetron cathodic sputtering in reactive mode [26, 27]. For the visualisation tests, the lower specimen was coated with a $1 \mu\text{m}$ thick $\text{MoS}_{1.6}$ coating; the upper specimen was a non-coated sapphire transparent first body. The maximum Hertzian pressure, which was 1 GPa for the "blind" tests, was therefore 0.5 GPa for the visualization tests with the same load. A frequency of 10 Hz was used for blind tests and 0.1 Hz for visualisation tests.

Although the first bodies were different, the "blind" and visualisation tests were nonetheless comparable, because the results in terms of flows and rheology were not significantly influenced by the nature of the materials composing the first bodies (steel or sapphire) when taking into account the test conditions chosen [17]. These visualisation tests enable the reconstitution of the third body flows brought into play with the opaque first bodies, i.e. during the "blind" tests, by reducing the role played by imagination.

The variable parameters were the temperature (297 K, 223 K, 173 K and 123 K) and the gaseous atmosphere in the test chamber, a dry mixture of nitrogen and oxygen gas

(80% N₂ + 20% O₂) noted "N₂+O₂", and a pure nitrogen gas, noted "N₂". The temperature of a specimen was measured with a copper-constantan thermocouple in the bulk at 1 mm under the surface. Due to the low temperature, the tests were performed in a gas environment with very little amount of water vapour to avoid frost. Ultra-pure compressed gas were used. Both gaz, "N₂+O₂" and "N₂", contained less than 0.5 ppm-mol of H₂O. Before each test and cooling stage, the chamber was first blasted with "N₂" at room temperature for 30 minutes, thus removing air from the chamber.

Both friction tests and lifetime tests were performed for the given test conditions. "Blind tests" of different durations were performed on different samples, because stopping and assembling first bodies of the friction pair would have involved repeated running-in. Lifetime was defined when the friction coefficient reached a value over 0.2.

2.3. Third body concepts

This work uses a mechanical approach based on the third body concept [28, 29]: the tribological triplet, the tribological circuit [30] and the rheology of third body. The third body is the volume of material that separates the bodies in contact (called first bodies) while accommodating most of their difference in speed. "Elementary entities" are detached from one or the two first bodies, then agglomerate. The solid particles can be trapped in the contact or be definitively lost for the contact. ~~So the third body is an "operator" that separates the first bodies and transmits the load between the first bodies while accommodating the greater part of their difference in speed.~~

The flows of the third body, constituting the tribological circuit, are first defined for an elementary contact. The internal source flow Q_s^i corresponds to the detachment of particles due to superficial tribological transformations [31], cracking, adhesion, etc. It leads to the formation of the natural third body. In this paper, the coating is the main source of the third body. This formation leads to the identification of three steps of the contact's life, which are

the phases of conception, birth (or running-in period) and actual life of the third body and, by extension, of the contact [30]. The internal flow Q_i is the flow of the third body circulating between the 1st bodies. The external flow Q_e is the flow of the third body that escapes from the contact. It splits into a recirculation flow Q_r and a wear flow Q_w . Recirculation flow Q_r is composed of the third body reintroduced into the contact, carried along, for example, by one of the first bodies and which will contribute once again to the velocity accommodation of the contact. Wear flow Q_w , is composed of the third body that has been definitively ejected from the contact and can no longer participate in velocity accommodation. Measuring these flows requires the technique of in-situ tribometry. It allows for real time observation of third body behaviour through the use of a transparent sample. But three-dimensional visualizations raise photographic and image processing problems [17, 32]. Consequently, the flows are only estimated according to a relative scale (from a “very, very low” value to a “very high” value). This qualitative flow scale uses the * symbol.

The description of the accommodation of relative velocities uses five sites (S) and four modes (M). In this mechanical approach, the volume part (noted S_3) of the third body is distinguished geometrically from the “screens” (noted $S_{2,4}$). S_3 , whose thickness can reach about ten micrometers, is constituted by the solid particles circulating in the contact ~~and forming a “more or less continuous film”~~. $S_{2,4}$, whose thickness is usually < 50 nm, is formed by surface complexes on the surfaces of the bodies in contact. S_1 and S_5 are the first bodies in contact. The four modes of accommodation described how accommodation occurs: elastic deformation (M1), normal rupture (M2), shearing (M3) and rolling (M4). One site can be associated to several modes.

Since it cannot be measured, the rheology of the third body is evaluated on the basis of its “cohesion” and its “ductility” [17, 33]. These two properties are determined by

observations of the third body (optical and scanning electronic microscopy) and are related to its morphologies and texture, present in most cases in each of the contact conditions at a given scale. ~~The word “texture” is not used in the metallurgical sense but refers more classically to the orientation of the third body layers or the third body particles, and their size, shape and arrangement.~~ The figure 2 illustrates physically the cohesion and the ductility of the third body formed from the $\text{MoS}_{1.6}$ coating at room temperature [17], which could be considered as a chart for this work. The term cohesion has been chosen by analogy with the cohesion of powder mixtures [34]. Examination on the same scale of observation shows that a third body of very low cohesion will be very powdery and composed of small particles in comparison to a cohesive third body. Since a highly cohesive third body tends to be very compact, it also appears to be more "continuous" [33]. The qualitative scale of this cohesion is symbolised by +. The term ductility has been chosen by analogy with the plastic flow characteristics of metals. The ductility expresses the ease with which the third body spreads and extrudes in the contact. Thorough analysis of the texture (spreading, continuous or discontinuous film, etc.) shows that the flow of the third body occurs with increasing difficulty, corresponding to the extent to which the boundary of its volume is "scalloped" [33]. ~~This texture analysis can be carried out by image analysis. The notion of ductility indirectly incorporates the adhesion of the third body to the first body. The qualification of ductility is calculated from the dimension of the third body islets activated.~~ Low ductility means that the third body spreads little and only covers a small area of the contact. Conversely, high ductility means that the third body spreads easily and covers a large part of the contact area. The qualitative scale of ductility uses the # symbol.

The relative values of the cohesion and the ductility are represented in the text by their symbols, respectively +, #, whose number (max=6 symbols) is proportional to each of these qualitative values. They are evaluated at a given scale and correlated with friction

values. It is clear that despite the unsatisfactory nature of this phenomenological procedure, it is much more preferable than using static characterisations that are even further from reality [33, 35]. Moreover, rheology is a mechanical concept that takes into account the contribution of physical chemistry.

This work is essentially a phenomenological work, as tools for quantifying solid third body flows and rheology are still at development stage at present [36-38].

Friction value, wear and lifetime are determined on the basis of the solid third body formed from the bodies in contact and circulating between them [30, 33]. More specifically, this is done via the flows of the third body and the rheology activated in the contact. This is the case with solid lubrication where the third body stems mainly from only one of these bodies.

2.4. Reconstitution of the contact life

The reconstitution of the contact dynamics was carried out by comparing post-mortem analyses, i.e. after the "blind" or visualization tests, and in-situ analyses, i.e. during visualization tests. In-situ dynamic observations (visualization tests) were video-recorded on at 25 frames/s (VHS). The visualization tests highlighted the evolutions of the different third body flows; it also helps to determine the site and mode of velocity accommodation. Different post-mortem analyses of the contact zones of both the opaque and transparent first bodies and their comparison were carried out after experiments, using different tools: Optical Microscopy, Scanning Electron Microscopy (SEM) coupled with Energy Dispersion X-ray analysis (EDX), and Optical Profilometer.

3. Results and discussion

3.1. General friction and lifetime results

In both atmosphere conditions, " N_2+O_2 " and " N_2 ", the friction log (figure 3) showed that the tests were carried out in gross slip regime. ~~The friction coefficient and lifetime were reproducible within the experimental error.~~ In oxidising atmosphere, " N_2+O_2 ", the friction curves (figure 4) showed a quite short running-in period for all temperatures and atmosphere, except at 123 K for which it increased drastically immediately at the beginning of the test. Nevertheless the friction coefficient value increased when temperature decreased (Table 2) under oxidising atmosphere. Under nitrogen, the friction coefficient was the same at both temperature 123 K and 297 K.

Lifetime showed significant temperature dependence under oxidizing atmosphere (Table 2). At 123 K, lifetime was about 10 times lower than at room temperature. Under nitrogen " N_2 " the lifetime had not been reached during the experiments ($> 40\,000$ cycles).

T(K)	123	173	223	297
Friction coefficient after 5000 cycles	0.11	0.04	0.035	0.03
Lifetime (10^3 cycles)	5.3	15.5	32.	47.6

Table 2: Friction values and lifetime function of temperature ("blind" tests) under " N_2+O_2 ".

3.2. Effect of temperature under oxidising atmosphere " N_2+O_2 "

3.2.1 Temperature of 297 K

Figure 5 shows the post-mortem static observations by optical microscopy of each of the first bodies tested at 297 K. The Hertzian contact area is schematised by a dotted line.

The contact can be divided into three zones: 1- the entry zone, the leading/trailing (right/left) edge in reciprocating kinematics, located in front of the contact; 2- the internal zone, initially defined by Hertz's theory; and 3- the lateral zones, located on either side of the internal zone. Each of these zones contributed more or less to load-carrying and to the difference of velocity accommodation between the first bodies. At a smaller scale of observation, the active flows of the third body in each of these zones can be localised in areas of a few square micrometres on the surfaces, called third body islets. These can be detected by SEM and EDX analysis (figure 6). The third body constituting these islets is characterized chemically.

Analysis of the third body, composed of a sulphur and molybdenum base, indicated that Q_s^i stemmed from the $MoS_{1.6}$ coating while the presence of oxygen indicated interaction with the atmosphere. Detection of iron on the coated first body indicated consumption of the coating. The density (in number) of islets was over 20 and their surface area varied from about 10 nm^2 to a few thousand μm^2 . This indicated that the third body formed was ductile (#####). Table 3 shows that the third body was cohesive at 297 K. Profilometry provides information of the thickness of the third body in the entry and lateral zones and thus on its volume.

T (K)	123	173	223	297
SEM image ↑↓ d
Cohesion	++++	++++	++++	++++
Ductility	##	###	####	#####

Table 3: Third body rheology in " N_2+O_2 ", function of temperature

Three images from a video film were extracted at 100, 500 and 1000 cycles (figure 7).

The dynamic observations gave a view of the flows activated and the evolution of the third body. The flows are represented on the figures by arrows **which** thickness is proportional to the value of the flow. Q_s^i was not very high (***) in an atmosphere of "N₂+O₂" and was located during the birth phase in the three zones of the contact. In the entry zone the volume of third body formed was the highest. During the actual life of the contact, Q_s^i was mainly activated in the entry zone. The direct exchanges between the coating and the atmosphere were only allowed in the entry zone because of the magnitude d . The entry zone was constituted by an accumulation of a cohesive third body (++++). Furthermore, the direct exchanges between the third body and the atmosphere were only allowed in the entry zone. When the third body passed from the entry zone to the internal zone, Q_r is activated (***). The third body, present in the internal zone, flowed in the **shape form** of islets. Their surface density and their surface area were similar to the post mortem analysis. The third body formed was ductile (####). These islets of third body relayed each other due to the deformations caused by the flow in the contact. This indicated that Q_i was activated (**).

3.2.2 Temperature of 123K

Figure 8 shows, the post-mortem static observations by optical microscopy of each of the first bodies at 123 K. The contact can also be divided into three zones: 1- the entry zone, 2- the internal zone, 3- the lateral zones.

The entry zone was constituted by the accumulation of a cohesive third body (+++++) during the contact's lifetime (table 3). This third body was composed of sulphur and oxygen, thus indicating interaction with the atmosphere (figure 9). Therefore **magnitude amplitude** d allowed direct exchanges between the third body and the atmosphere; these exchanges only occurred in the lateral and entry zones.

In the internal zone, very few islets composed of sulphur were detected on the convex first body by EDX analysis (figure 9); moreover, no iron was detected on the flat first body (figure 10). This means that hardly any third body was present in the internal zone and that flows occurred in the form of a very small number of activated islets. It appeared that the ~~density in~~ number of the latter was lower than thirty and their surface areas varied from about 10 nm² to a few dozen μm². This indicates that Q_i was hardly activated at all (**). Profile measurements (figure 11) coupled with EDX analysis of the first bodies at 5000 cycles showed a high quantity of third body debris. The ductility of the third body formed was poor (# #) (Table 3), which explained that a very small amount of third body entities passed from the entry zone to the internal zone, and thus there was very little activation of Q_r (*). Consequently, the third body (S₃) in the inlet zone contributed to load-carrying ~~##~~ ~~123 K~~. But it did not replenish the contact and so it was not reused to lubricate the contact. The third body was quickly pushed out of the entry zone and thus ejected from the contact (figures 8, 9). This indicated a high volume of ejected third body and thus considerable activation of Q_w (*****). The main accommodation zones were therefore the lateral zones and the entry zone, in the third body volume S₃.

At 123 K, three images from a video film were extracted at 100, 500 and 1000 cycles (figure 12) under oxidising atmosphere.

Q_sⁱ was high (*****) and was located during running-in period in the lateral zones (figure 9a), thus the volume of third body formed was quite high. During the actual life of the contact, Q_sⁱ was still activated in the lateral zones and also activated in the entry zone.

At 123 K lifetime was about 5000 cycles (Table 2). Iron was identified in the entry (figure 10) and lateral zones on the flat first body and the profile measurements showed a valley depth of about 1 μm (figure 11), which was the thickness of the coating. Thus the

coating was totally consumed; therefore Q_s^i was regularly activated during the test. This can be explained by the steady activation of Q_w coupled with the non-activation of Q_i . Therefore friction coefficient increased gradually.

3.2.3. Intermediary temperatures

When decreasing the temperature gradually, under " N_2+O_2 ", no critical temperature was noticed and no threshold effect for friction values was observed.

In an " N_2+O_2 " atmosphere at the 4 temperature conditions, Q_s^i was activated throughout the life of the contact. SEM observations of the coated planes and profile measurements and dynamic observations at the 4 temperatures confirmed that Q_s^i was activated in the entry zone and in the lateral zones. At 173 K (figure 13), 223 K (figure 14), 297 K, observations of the first bodies after 10000 cycles showed that the entry zone was constituted by the accumulation of a cohesive third body. However, this accumulation was greater at lower temperature. Thus the volume of the third body formed was higher at 123 K than at 297 K. Therefore Q_s^i increased as temperature decreased (*** at 297 K, ***** at 123 K).

The third body was also composed of sulphur and oxygen at the 4 temperatures, thus indicating interaction with the atmosphere and exchanges between the third body and the atmosphere occurred in the entry zone and the lateral zones. Dynamic observations and static observations showed that the third body passed less from the entry zone to the internal zone as temperature decreased. This was due to the decrease of the third body's ductility and the increase of its cohesion at lower temperature (Table 3). In " N_2+O_2 ", the third body showed a higher brittleness when the temperature decreased. As the temperature decreased the ductility of the third body gradually decreased, from ##### at 297 K to ## at

123 K. Its cohesion increased gradually, from + + + + at 297 K to + + + + + at 123 K (Table 3).

The accumulation of third body in the entry zone increased gradually as temperature decreased. The third body accumulated in the entry zone contributed to load-carrying (S_3) at the 4 temperatures. However, the accumulated third body at the entry zone that contributed to Q_r at 297 K, did not contribute to Q_r at 123 K. Thus the activation of Q_r fell as the temperatures decreased (** at 297 K, * at 123 K). After 10 000 cycles, the ~~volume~~ amount of third body ejected from the contact was higher at 123 K than at 297 K. In the same time decreasing temperature reduced the density of islets in the internal zone. At room temperature the third body, present in the internal zone, flowed in the form of thin islets (mainly $S_{2,4}$). This indicated that Q_i was activated (****). At 123 K no islets were observed; this indicated the reduction of Q_i at 123 K compared to the case at 297 K (** at 123 K). Thus, as temperature decreased, the internal zone contributed less and less to velocity accommodation; the accommodation zone became mainly the lateral and entry zones. Furthermore Q_s^i was more activated at low temperature in the entry zone. Therefore Q_w was highly activated at low temperature. That explained the decreased lifetime when temperature decreased.

At 173 K it has been noticed that the friction coefficient reached a mean value of 0.06 during 1000 cycles. This was explained by the formation of third body at the entry zone which acted as a reservoir during few number of cycles. The rheology of third body allowed its trapping. Q_s^i was thus partially interrupted. The friction coefficient decreased to a mean value of 0.04 during few thousand of cycles. Nevertheless the third body was gradually pushed out of the entry zone and thus ejected from the contact. Q_s^i was activated anew.

3.3. Effect of temperature under "N₂" atmosphere

During the actual life of the contact, the lateral zones and the entry zone on the two first bodies remained empty of third body (Figures 15, 16). Therefore Q_r was not activated (*). No ejected third body particles were observed. Thus Q_w had not been activated (*). The accommodation zone was localized in the internal zone whose area remain constant and close to the Hertz area (figure 16). The third body, whose thickness was much less than 200nm, was organized in the form of few "tapered strips", drawn in the direction of the ~~movement~~ motion (Table 4), with a length of 100 μm , which is the total stroke (2x50 μm) of motion (figure 15). They constituted thin third body islets and thus contributed to Q_i . The third body formed was not very ductile (#). The specific surface density of the islets was much higher than the one under oxidising atmosphere, and their surfaces areas were very small, from about 10 nm^2 for a contact area of 50.10^{10} nm^2 . No oxygen was detected so there were no chemical reactions with the atmosphere. The extent of the action of the third body was therefore much more reduced than under "N₂+O₂" atmosphere.

Under "N₂" atmosphere, the temperature did not affect the flows and the rheology of third body. That explains the friction coefficient was the same whatever the temperature was.

Temperature		123 K	297 K
SEM image	Sliding direction
Rheology	Cohesion	+	+
	Ductility	#	#

Table 4: Relative third body rheology of solid third body in "N₂", function of temperature

3.4 Effect of the atmosphere and temperature

This work highlighted a different behaviour in " N_2 " and in " N_2+O_2 " atmosphere. General results based on long-term experimental study [17] have shown that the action of physical chemistry via the atmosphere mainly determines the rheology of the third body during sliding. This is decisive for friction and wear results: in the case of " N_2 " atmosphere, the very fine thickness of the third body implies velocity accommodation within the "screens" ($\text{S}_{2,4}$), i.e. the physical-chemical scale is pertinent [39], friction is low and lifetime is improved. Increasing the effect of the bulk of the third body (S_3) involved leads to mechanical effects, and consequently more "rheological" effects. This occurs in the case of " N_2+O_2 " gas.

Dimigen et al [18] showed that non-stoichiometric layers MoS_x deposited by r.f. sputtering had a strong dependence of friction on the actual oxygen pressure during friction tests at ambient temperature in continuous kinematics; the hypothesis was that the non-saturated binding forces cause absorption of the ambient gas. Those tests have been done at oxygen partial pressure of $3 \cdot 10^{-7}$ and 10^{-4} Pa. A small fraction of oxygen could help low friction conditions [18, 40, 41]. In the case presented in this paper, the quantity of oxygen under " N_2+O_2 " were higher than in the cited studies. Nevertheless the exposure of $\text{MoS}_{1.6}$ coating to " N_2+O_2 ", before friction and during test, could allow formation of adsorbate layer with oxygen, as it was mentioned in [18, 42]. Additionally, the third body have been certainly oxidised during friction [40]. It should also be noted that $\text{MoS}_{1.6}$ coatings were deposited by d.c. magnetron sputtering; the films were microcrystalline with a (001)-preferred orientation and very dense [26, 27] which differed from the fibrous structure of r.f. sputtered coatings [26, 40, 43]. That could imply different oxidative reactivity [44] and thus different third body flows.

In the case of MoS_2 , recent results [41] at 297 K showed also that wear was more significant under air than under " N_2 ", because " N_2 " environment modified velocity

accommodation mechanisms. Indeed, low friction and low wear would be due to " N_2 " adsorption which formed surface complexes on the surface of the coating but also on the third body particles. Thus all potential oxidization sites were occupied [41]. Consequently the reactivity of the coating and the third body could be minimized or offset.

At 297 K, the friction behaviour of $MoS_{1.6}$ was different under " N_2 " and " N_2+O_2 " atmosphere. As the content of impurities, especially the H_2O content was the same in the " N_2 " and " N_2+O_2 " gas, the difference was attributed to a difference of surface reactivity linked to different surface complexes formed on the surface. That led to a different Q_s^i . The third body formed was also different: it was oxidised under " N_2+O_2 ". Thus the wear lifetime was modified significantly.

This difference still stands when the temperature decreased. In " N_2+O_2 " atmosphere, the volume of the ejected third body (Q_w) was much significant and Q_s^i was higher than in " N_2 " atmosphere, whatever the temperature was. SEM analysis showed that the trapped third body appeared to flow with more difficulty with the temperature decrease in " N_2+O_2 " atmosphere. Thus the temperature decrease modified the ductility of the third body. Unfortunately the mechanical properties are not yet properly characterised [45] even at ambient temperature. The fact that the modification of the ductility is due to a modification of the mechanical properties of the third body, with decreased temperature, remains an assumption.

In the case of " N_2 " atmosphere, the temperature decrease did not modified the friction behaviour of $MoS_{1.6}$ coating. On contrary, a study, performed by Hamilton et al. [7] on different MoS_2 based coatings (containing various metals or metal oxide) under nitrogen gas in the temperature range between - 80 °C and + 180 °C, under continuous kinematics, showed an increased friction with decreased temperature. But the coatings were different. That could imply different surfaces complexes, thus different surface reactivity.

Furthermore the third body formed from those coatings was different than the one formed from MoS_{1.6} coating. As a consequence the flows would be different in both studies and the equilibrium of the flows would also be different. The comparison of both studies is thus uncertain.

The question remains on the presence of ice or not during the low temperature tests. The sapphire, used during the visualization tests, allowed to see the surface of the coating during the cooling stage and during the tests. No modification of the aspect and the reflectivity of the pristine surface was noted. Furthermore in another study friction coefficient have been measured at different temperatures between ice layer and steel [46]. The friction stabilized for many cycles. Its sudden increase indicated the ice layer was worn away. During the tests with MoS_{1.6} coating, this trend was not observed. Those information suggested that the surface was not covered by a layer of ice or frost at the end of the cooling stage and during the tests at a macroscopic scale. Post mortem analysis of tracks after different number of cycles under "N₂+O₂" have shown a gradual consumption of the coating in the entry zone; the gradual increase of the friction coefficient was linked to a constant activation of Q_s^i . Nevertheless, as the H₂O content in the gas was less than 0.5 ppm, it may be possible that a small amount of water may be trapped in the coating porosities. During cooling the small quantity of water would be transformed into ice nano-crystallites and thus would be included in the third body formed. Under "N₂", Q_s^i was very low, so if ice nano-crystallites were trapped in the coating, their contribution to third body flows was negligible. On contrary, under "N₂+O₂", ice nano-crystallites may contribute to the third body flows. But the interpretation of the loss of ductility of the third body under "N₂+O₂" with the temperature decrease is still a matter of investigation. It may be due to the presence of ice crystallites in the third body, to the materials properties modification of the third body or both.

4. Conclusion

Friction and lifetime of a sliding contact "lubricated" by a solid third body formed in situ by an MoS_{1.6} coating were successfully evaluated at low temperature. These friction results are correlated with modifications of the third body's rheology and flows, which were determined by static and dynamic observations. Analysis of the results in the oxidizing atmosphere shows:

- under both ambient and low temperatures conditions the volume of the third body in the contact stems from a high source flow Q_s^i value,
- oxygen allows to form a sufficient volume of third body after which velocity accommodation occurs in its volume (S_3) and not only in the screens ($S_{2,4}$),
- the volume of the third body increases at the edge of the Hertzian zone as temperature decreases,
- no threshold effect could be seen from 123 K to 297 K.
- the contact's recirculation flow Q_r reduced considerably and Q_s^i increased as temperature was decreased, which explains the reduction of the contact's lifetime when changing from ambient temperature to 123 K (Table 5). At this temperature, the source flow Q_s^i is almost equal to the wear flow Q_w , leading to short lifetime. At 297 K the wear flow Q_w is reduced due to the activation of the recirculation flow Q_r and the internal flow Q_i .

In an nitrogen atmosphere, the friction value was low at 297 K and at 123 K.

At ambient temperature velocity accommodation, in " N_2+O_2 " (Table 5), was localised both in the Hertzian zone (internal zone), with activation of $S_{2,4}$, and at the edge of the Hertzian zone (entry zone and lateral zones), with activation of S_3 , whereas at 123 K it is almost exclusively localised at the edge of the Hertzian zone, with the activation of S_3 . The values of the tangential force, thus of the friction, stem from the localisation and activation

of $S_{2,4}$ and/or S_3 . Indeed, for an equivalent surface, the friction obtained by activating the screens ($S_{2,4}$) is generally inferior to that obtained during the activation of the volume of the third body (S_3).

Temperature (K)		123	297
Flows	Q_s^i	* * * * *	* * *
	Q_i	* *	* * * *
	Q_r	*	* *
	Q_w	* * * * *	* * *
	Equilibrium (tribological circuit)
Rheology	Cohesion	+ + + + +	+ + + + +
	Ductility	# #	# # # #
Accommodation zones		entry +	entry + lateral + internal
Accommodation sites		S_3	$S_3 + S_{2,4}$

Table 5: Synthesis of the results function of temperature; dry atmosphere " N_2+O_2 "

From a geometric point of view, the volume of the third body at low temperature is sufficient to separate the first bodies and impose macroscopic load-carrying zones at a scale greater than that determined by the deformations of the first bodies (Hertz), whereas at ambient temperature the deformations of the first bodies are of the same order of magnitude as the thickness of the third body. Consequently, these deformations are no longer negligible in defining the load-carrying zones. In other words, the macroscopic load-

carrying zones are determined by the third body at low temperature in " N_2+O_2 ", whereas they are determined by both the third body and the deformations of the first bodies at ambient temperature.

More generally, this tribological view makes it possible to approach sliding contact friction and lifetime successfully by taking into account the rheology and flows of the third body.

Acknowledgments

The authors would like to thank Georges Roche for the design of the specific cooling chamber and the US Air Force for its financial support (Grant no. AFOSR-90-0361).

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Figure captions

Figure 1: Schematic design of the test chamber

Figure 2: Rheology of third body - Scale of cohesion and ductility, from low to high [17]

Figure 3: Friction logs (tangential force F_t , displacement d , number of cycles), under the atmosphere " N_2+O_2 ", at 4 temperatures T , (a) 297 K, (b) 223 K, (c) 173 K, (d) 123 K

Figure 4: Friction coefficients under two different atmospheres at 4 temperatures

Figure 5: Static observation; $T=297$ K; Friction tracks after 10000 cycles, $d=\pm 50\mu m$, atmosphere of " N_2+O_2 ", (a) flat 1st body, (b) convex 1st body

Figure 6: EDX analysis of the convex first body; $T=297$ K, " N_2+O_2 ", after 5000 cycles; element distribution in white, (a) iron, (b) oxygen, (c) sulphur

Figure 7: Dynamic observations; $T=297$ K; Visualization test, $d=\pm 50\mu m$, atmosphere of " N_2+O_2 ", (a) 30 cycles, (b) 500 cycles, (c) 1000 cycles

Figure 8: Static observations after 5000 cycles; $T=123$ K, " N_2+O_2 ", (a) convex 1st body, (b) flat 1st body

Figure 9: EDX analysis of the convex first body; $T=123$ K, " N_2+O_2 ", after 5000 cycles; element distribution in white, (a) iron, (b) oxygen, (c) sulphur

Figure 10: EDX analysis of the flat first body; $T=123$ K, " N_2+O_2 ", after 5000 cycles; element distribution in white, (a) iron, (b) oxygen, (c) sulphur

Figure 11: profile measurement, after 5000 cycles at $T=123$ K under " N_2+O_2 ", (a) Convex first body, (b) Flat first body

Figure 12: Dynamic observations; $T=123$ K; Visualization test with the atmosphere of " N_2+O_2 ", (a) 100 cycles, (b) 500 cycles, (c) 1000 cycles actual life

Figure 13: Static observations after 5000 cycles; $T=173$ K, " N_2+O_2 ", (a) convex 1st body, (b) flat 1st body

Figure 14: Static observations after 5000 cycles; $T=223$ K, " N_2+O_2 ", (a) convex 1st body, (b) flat 1st body

Figure 15: Static observations - Friction tracks after 10000 cycles ($d = \pm 50\mu m$, atmosphere of " N_2 ") at 297 K

Figure 16: $T=130$ K; Visualization test with the atmosphere of " N_2 ", (a) 1000 cycles, (b) 9000 cycles actual life

Table captions

Table 1: Synthesis

Table 2: Friction values and lifetime function of temperature (“blind” tests) under “N₂+O₂”.

Table 3: Third body rheology in "N₂ + O₂", function of temperature

Table 4: Relative third body rheology of solid third body in N₂, function of temperature

Table 5: Synthesis of the results function of temperature; dry atmosphere "N₂ + O₂

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Figure 4: Friction coefficients under two different atmospheres at 4 temperatures

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