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Titre	**HTRE – Deliverable 41 of WP5** (ref : 05/12-D-5-3-1-1/2-b) **Results of elementary tests**
Auteur(s)	CACHON L.

Abstract :
Within the framework of the European Program HTR-E of the WP5, this document puts the emphasis on the CEA contribution of the deliverable D41 which aims to make a set of elementary tests in helium environment for CGR applications. The tests were performed on different materials like on like contact :
HR230, CERMET (Cr3C2 – NiCr), Zirconium coatings (ZrO2-Y2O3 / NiCrAlY and 75% (ZrO2-Y2O3) + 25% CaF2 / NiCrAlY, and SiC (PVD). Just the CERMET coating allowed the realization of the 5000 required cycles at 700°C and 5 MPa of contact pressure, with a low wear rate (0.008µ/cycle) and a coefficient of friction lower than 0.3.

Résumé :
Dans le cadre du programme européen HTR-E WP5, ce document présente la contribution du CEA dans le livrable D41, qui consiste en la réalisation de tests élémentaires de tribologie en atmosphère représentatives des RCG. Les tests ont été réalisés sur différents matériaux en contact homogène : HR230, CERMET (Cr3C2 – NiCr), revêtements de zircone (ZrO2-Y2O3 / NiCrAlY et 75% (ZrO2-Y2O3) + 25% CaF2 / NiCrAlY, et SiC (PVD). Seul le revêtement CERMET a permis la réalisation des 5000 cycles requis à 700°C et 5 MPa de pression de contact, avec un taux d'usure très bas (0,008 µm/cycle) et un coefficient de frottement inférieur à 0,3.

Note technique vérifiée par F. WITTERS F. Wit

Mots clés	GCR, tribology, Zirconium, CERMET, SiC

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REFERENCES :

[1] Deliverable 40 of WP5 : proposal of elementary tests

[2] C. FALCAND, « Minutes of WP5 – 7th meeting for "Tribology in Helium Environment », Document N°: HTR-E-05/06-M-5-0-7

Revision object :

Add of NNC comments with the CEA Answer.

Contents

1. INTRODUCTION.

Within the framework of the European Program HTR-E of the WP5, this document puts the emphasis on the CEA contribution of the deliverable D41 which aims to make a set of elementary tests in helium environment for CGR applications.

Based on the previous deliverable, D40 [1], the tests program is summarized in the table 1.

N°	Component / contact	Environment	Material 1	Material 2	Temper. (°C)	Load (MPa)	Covered length (m)	Velocity (cm/s)	Tribometer FANP	Tribometer CEA	Remark
2	Reference test	Pure He	HR230	HR230	500	5	200	1			
4	Liner / Liner	Pure He	HR230 + ZrO_2/Y_2O_3	HR230 + ZrO_2/Y_2O_3	500	5	200	1			5MPa = limit load for liner, 900°C for PBMR, same procurement
6			HR230 + ZrO_2/Y_2O_3	HR230 + ZrO_2/Y_2O_3	900	5	200	1			
8		Pure He	HR230 + ZrO_2/CaO	HR230 + ZrO_2/CaO	500	5	200	1			
10			HR230 + ZrO_2/CaO	HR230 + ZrO_2/Y_2O_3	900	5	200	1			
12	Liner / Liner	Pure He	HR230 + SiC	HR230 + SiC	500	5	200	1			
14			HR230 + SIC	HR230 + SIC	900	5	200	1			
16		Pure He	HR230 + Cr_3C_2	HR230 + Cr_3C_2	500	5	200	1			
18			HR230 + Cr_3C_2	HR230 + Cr_3C_2	750	5	200	1			
20	Internals / vessel or Internals / block support	Pure He	9%Cr	9%Cr	250	50	200	1			
22			9%Cr	"	500	50	200	1			
24		Pure He	9%Cr + TiC	9%Cr	250	50	200	1			
26			9%Cr + TiC	"	500	50	200	1			
28			9%Cr + WC/NiCr	9%Cr	250	50	200	1			

A

N°	Component / contact	Environment	Material 1	Material 2	Temper. (°C)	Load (MPa)	Covered length (m)	Velocity (cm/s)	Tribometer FANP	Tribometer CEA	Remark
2	Reference test	He + impurities	HR230	HR230	500	5	200	1			
4	Liner / Liner	He + impurities	HR230 + ZrO_2/Y_2O_3	HR230 + ZrO_2/Y_2O_3	500	5	200	1			5MPa = limit load for liner, 900°C for PBMR, same procurement
6			HR230 + ZrO_2/Y_2O_3	HR230 + ZrO_2/Y_2O_3	900	5	200	1			
8	Liner / Liner	He + impurities *	HR230 + ZrO_2/CaO	HR230 + ZrO_2/CaO	500	5	200	1			
10			HR230 + ZrO_2/CaO	HR230 + ZrO_2/CaO	900	5	200	1			
12	Liner / Liner	He + impurities	HR230 + SiC	HR230 + SiC	500	5	200	1			
14			HR230 + SiC	HR230 + SiC	900	5	200	1			
16	Liner / Liner	He + impurities	HR230 + Cr_3C_2	HR230 + Cr_3C_2	500	5	200	1			
18			HR230 + Cr_3C_2	HR230 + Cr_3C_2	750	5	200	1			
20	Internals / vessel or Internals / block support	He + impurities	9%Cr	9%Cr	250	50	200	1			
22			9%Cr	"	500	50	200	1			
24		He + impurities	9%Cr + TiC	9%Cr	250	50	200	1			
26			9%Cr + TiC	"	500	50	200	1			
28			9%Cr + WC/NiCr	9%Cr	250	50	200	1			

B

Table 1 : Tests program for D41 (A- in pure He, B- in He + impurities).

Taking in account the delay to have the specifications of the atmosphere « He+impurities », and to finalize our alumina exchanger [2], all tests were performed in pure He.

2. CEA HE TRIBOMETER.

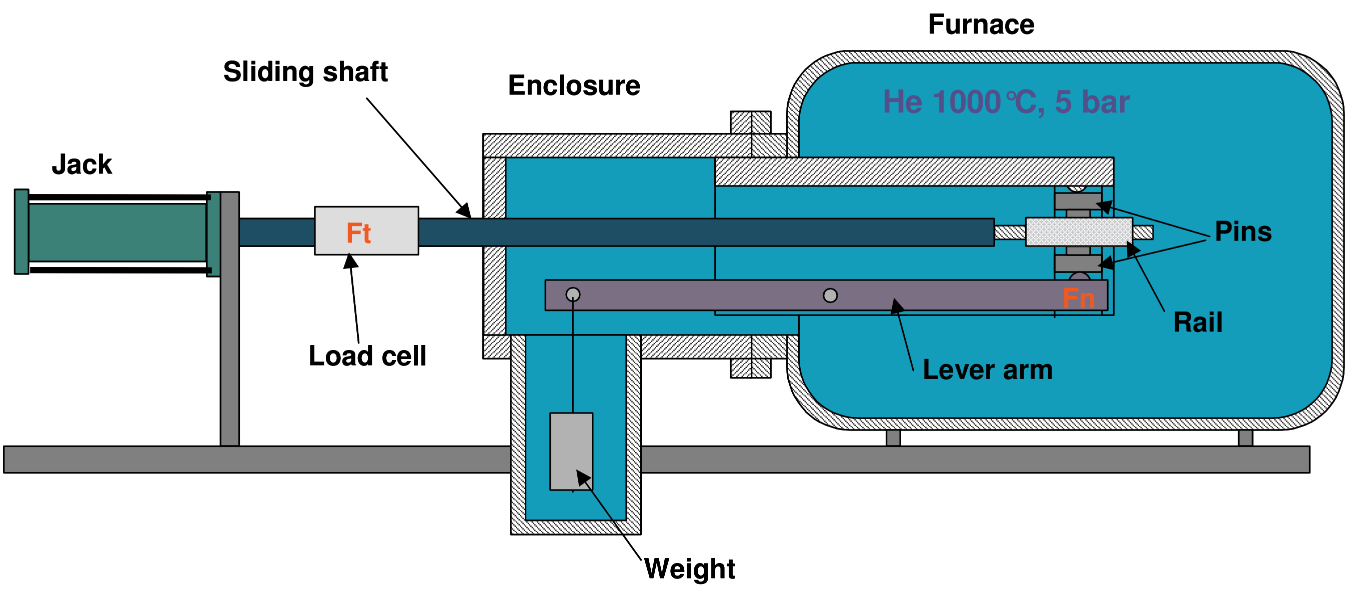
"He tribometer" is a "pins on rail alternative sliding" tribometer. This apparatus is divided in two parts :

- The mechanism which operates the load and the motion of the samples (2 pins and 1 rail),
- The furnace which operates the temperature and the pressure of the running atmosphere.

With a normal quasi-static load (F_N) applied to the pins, a tangential load (F_T) is needed to move the rail. The coefficient of friction, μ, is calculated from F_N and F_T as following :

$$\mu = \frac{|F_T|}{2 \cdot F_N}$$

The tangential load (F_T) is applied by a Jack via the sliding shaft. The normal quasi-static load (F_N) is applied with a weighting device (lever arm and weight).



Samples

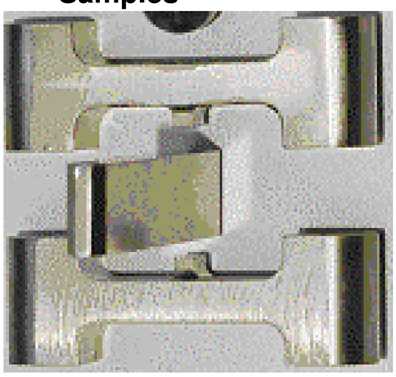


fig. 1 : He tribometer.

The major characteristics of He tribometer are :

- Plane/plane contact (0,5 cm²),
- Alternative motion,
- load : 0 – 20 MPa,
- Amplitude : up to 20 mm, Frequency : up to 6 Hz.
- Environment parameters :
  - Temperature up to 1000°C,
  - He + impurities

3. RESULTS.

In the next paragraphs, the results are presented as followed :

- Material description,
- Experimental parameters,
- Working atmosphere,
- Wear,
- Coefficient of friction,
- Discussion.

The objective of the tests is to perform 5000 cycles (≈200 m) without excessive damages and with coefficient of friction lower than 0.5.

3.1 HR230 like on like.

3.1.1 Material description

HAYNES® 230™ alloy is a nickel-chromium-tungsten molybdenum alloy (table 2) that combines excellent high temperature strength, outstanding resistance to oxidizing environments up to 1149 °C for prolonged exposures, premier resistance to nitriding environments, and excellent long-term thermal stability. It is readily fabricated and formed, and is castable. Other attractive features include lower thermal expansion characteristics than most high-temperature alloys, and a pronounced resistance to grain coarsening with prolonged exposure to high-temperatures.

Taking into account these physical and mechanical characteristics, this material is foreseen to mechanical used in high temperature, i.e. the mechanical part of the tribometer working up to 1000°C is made with HR230.

Nominal Chemical Composition, Weight Percent

Ni	Cr	W	Mo	Fe	Co	Mn	Si	Al	C	La	B
57ª	22	14	2	3*	5*	0.5	0.4	0.3	0.10	0.02	0.015*

*Maximum ªAs balance

table 2 : HR230 composition.

3.1.2 Experimental parameters.

The experimental parameters are :

- Temperature of 450℃,
- Contact pressure of 5MPa,
- Motion amplitude of 18 mm,
- Speed of 10 mm/s,
- Atmosphere : « pure He » (Tribometer feeding by Helium GC).

3.1.3 Working atmosphere.

During the test, the composition of the gas is well known and determined by sampling and chromatography analyses (table 3).

Samplings	O2 (ppm)	N2 (ppm)	CH4 (ppm)	CO (ppm)	CO2 (ppm)	H2O (UA)
O cycle 20℃	87	261	nd	nd	40	
After 1014 cycles at 500℃	3	134	32	37	4315	4.4

Table 3 : Result of chromatography analyse after gas sampling.

3.1.4 Wear.

After 2600 cycles, and the removal of the samples, a macroscopic observation of the contact (figure 2) show a wear essentially due to adhesion with a little part of abrasion occurred by wear debris (work hardened) harder than the massive materials.

figure 2 : Friction surface of HR230 after 2600 cycles at 450°C under 5 MPa of contact pressure

SEM observation show in more detail the surface topography, and X analyses highlight the different composition areas on the surface (figure 3) : dark areas have a higher level of oxygen, showing the presence of Ni and/or Cr oxides (Cr_2O_3, NiO or $NiCr_2O_4$).

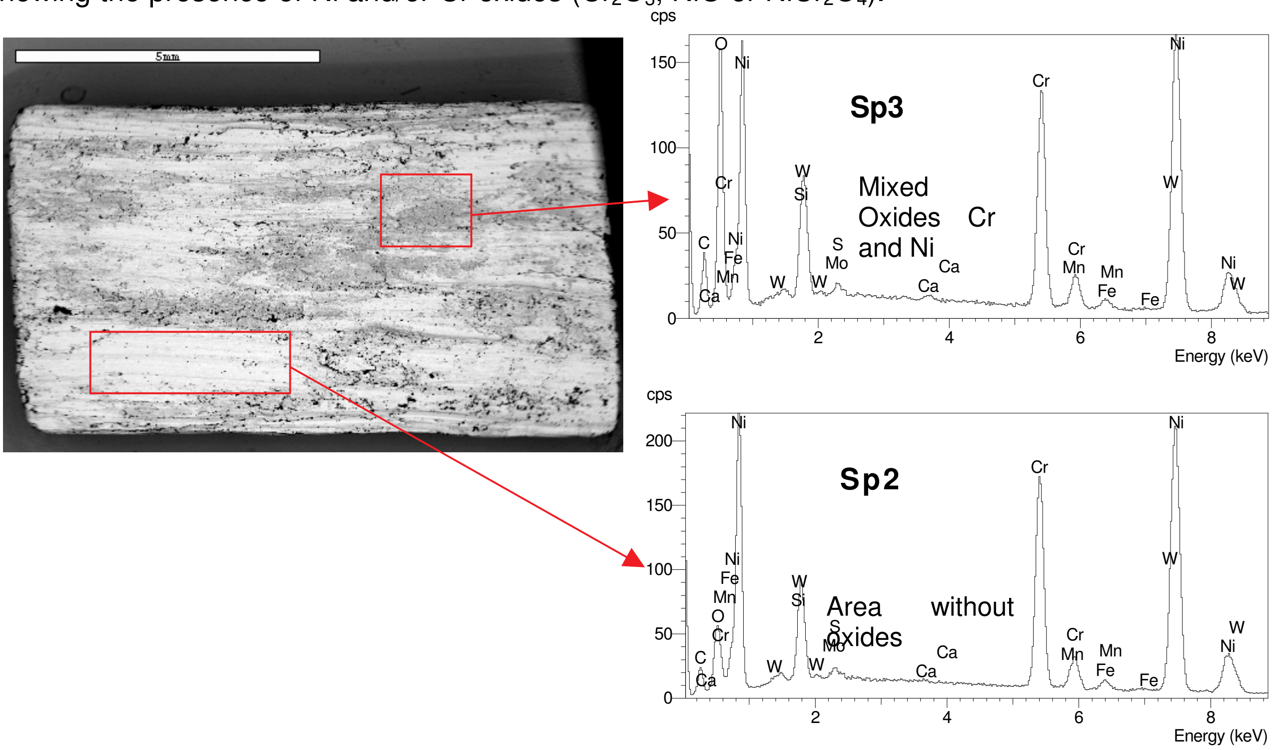


figure 3 : SEM and X analyse on HR230 samples.

3.1.5 Coefficient of friction.

The evolution of coefficient of friction during the 2600 cycles is show in figure 4. Up to 500 cycles, the mean coefficient of friction swing between 0.4 and 0.6. The perturbation of coefficient of friction during one cycle is equally higher, about ±0,22, than after 500 cycles which is about ±0,15.

After 500 cycles, the mean coefficient of friction is 0.42±0,15 and relatively stable up to 2600.

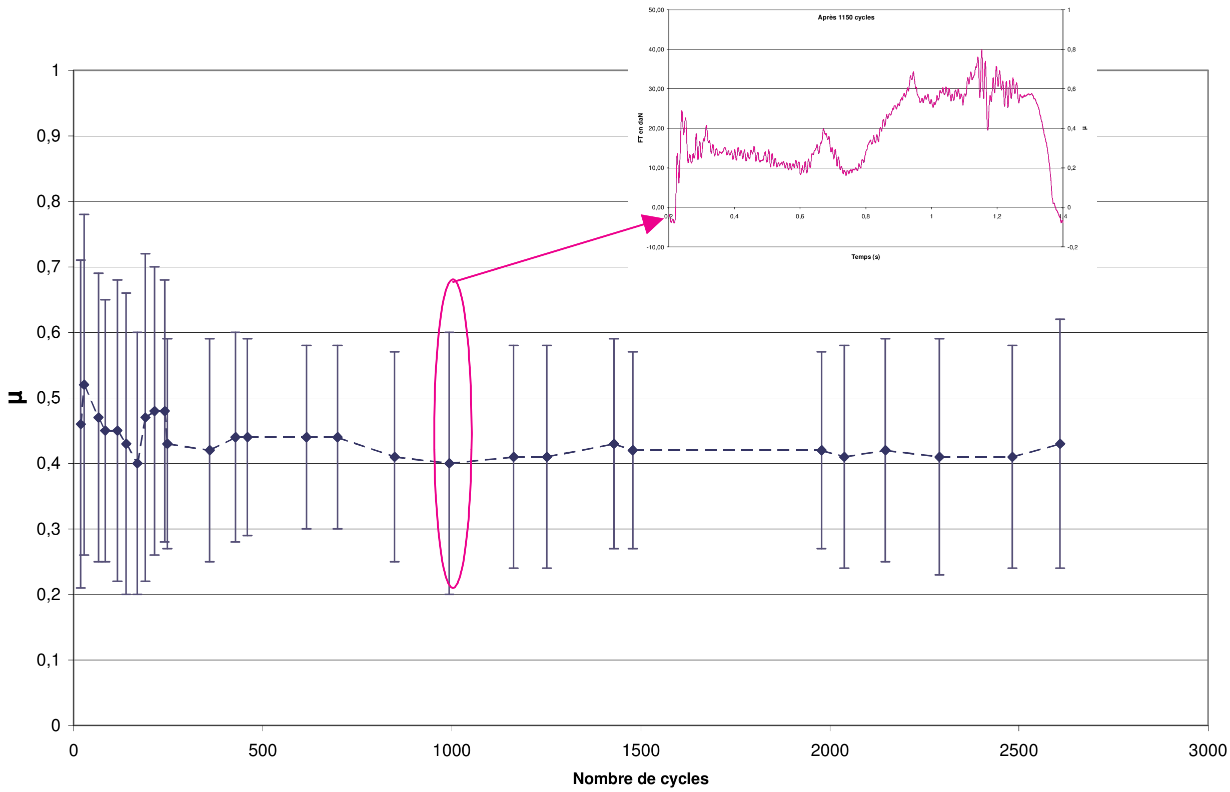


Figure 4 : Coefficient of friction evolution during cycles.

3.1.6 Discussion.

The HR230 is a metallic material, so very sensitive to adhesion in tribological solicitation. This sensitivity increases with the temperature. It is well know that for metallic materials, a critical temperature don't have to be exceeded to avoid severe adhesion. For Nickel base alloys, this critical temperature is about 0,4 x Tf, with Tf fusion temperature. So this critical temperature is about 500°C. Taking in account this characteristics, the results obtained in GCR environment shown that HR230 has a correct tribological behavior, without excessive damages.

3.2 ZrO_2-Y_2O_3 / NiCrAlY / HR230 like on like

3.2.1 Material description

The Zirconium coating (table 3) is obtained by classical PLASMA process (low pressure) on each friction surface of the samples realized in HR230. The procedure is as follow :

- Sand blasting of the surfaces to be coated,
- Coating of the NiCrAlY under layer, with a thickness of about 100 µm,
- Coating of ZrO_2-7%Y_2O_3 with a thickness of 300 µm,
- Regrinding of the surface in order to obtain a total thickness of the coating of about 250 µm.

ZrO_2	HfO_2	Y_2O_3	Al_2O_3	Fe_2O_3	SiO_2	TiO_2	CaO	U + Th	Other Oxides
90.00	1.80	7.50	0.08	0.02	0.25	0.10	0.02	0.03	<1.00

tableau 3 : Zirconium Yttried (ref :TSP#204F) coating composition.

3.2.2 Experimental parameters.

The experimental parameters are :

- Temperature of 800°C,
- Contact pressure of 5MPa,
- Motion amplitude of 18 mm,
- Speed of 10 mm/s,
- Atmosphere : « pure He » (Tribometer feeding by Helium GC).

3.2.3 Working atmosphere.

During the tests, the composition of the gas is well known and determined by sampling and chromatography analyses (table 4).

Samplings	O2 (ppm)	N2 (ppm)	CH4 (ppm)	CO (ppm)	CO2 (ppm)	H2O (UA)
O cycles 20°C	87	261	nd	nd	40	
After 1014 cycles at 500°C	3	134	32	37	4315	4.4

Table 4 : Result of chromatography analyze after gas sampling.

3.2.4 *Wear.*

After 1500 cycles and the removal of the samples, an observation of the friction surfaces shows a too high wear of the coating, fetches up to crack of one of the surfaces of the rail (figure 5). The main accommodation way is “abrasion”, with the formation of debris like a powder.

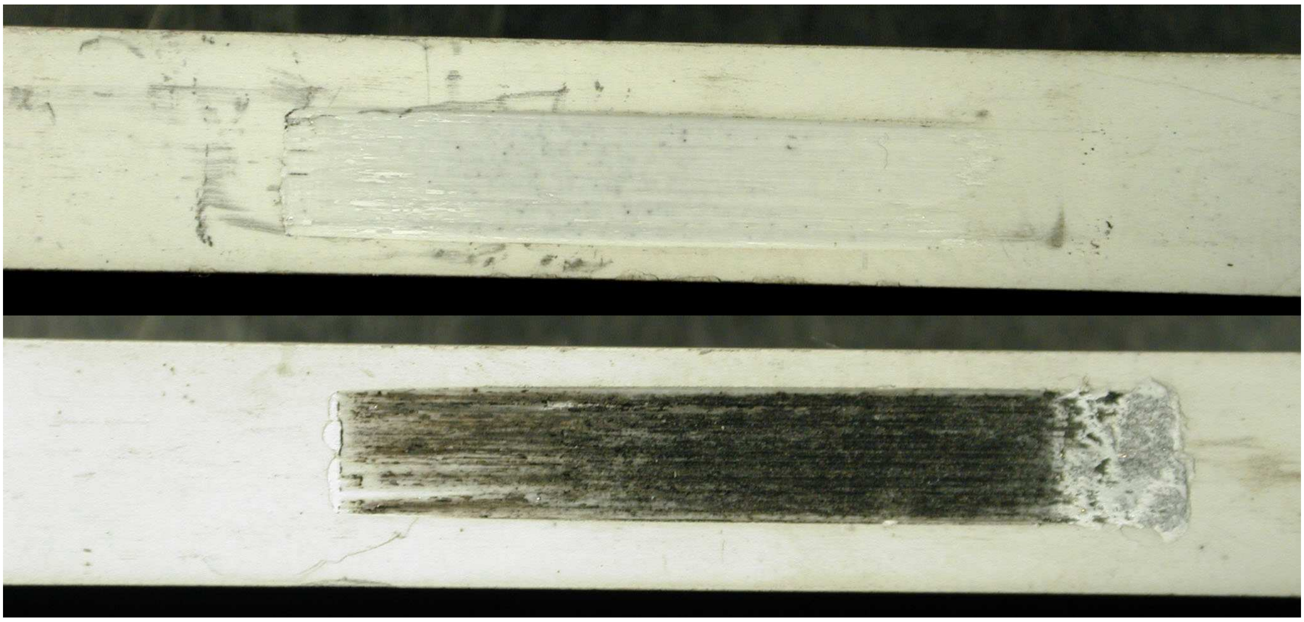


figure 5 : Friction surfaces of the rail coated by Zirconium after 1500 cycles under 800°C and 5 MPa of contact pressure.

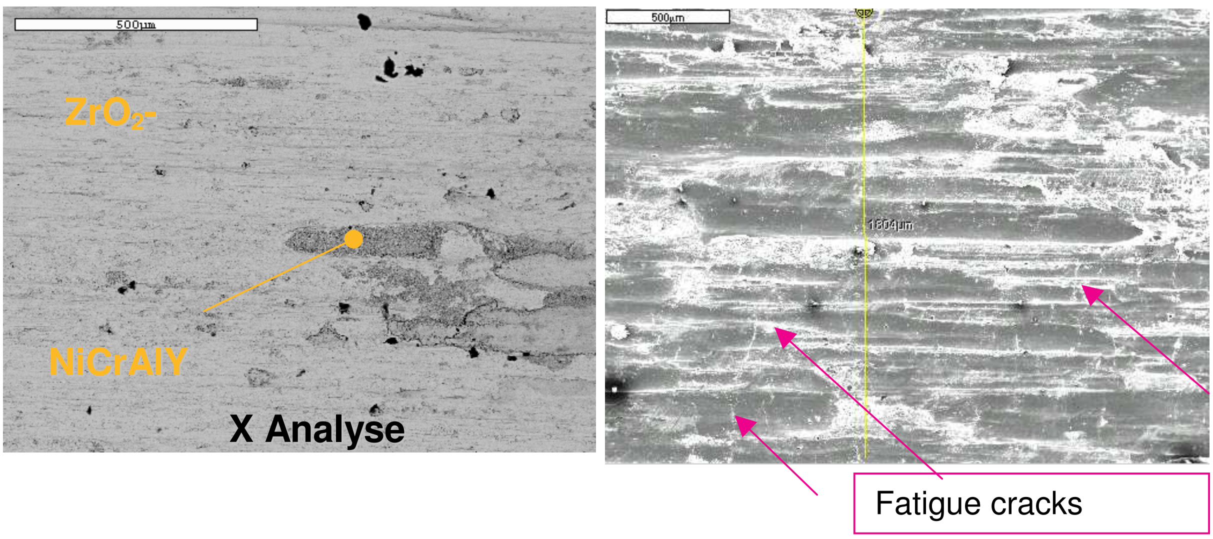


Figure 6 : SEM observation and X analyses performed on Zirconuim coating.

SEM observation and X analyses (figure 6) confirm the first observations made after 1500 cycles : the coating is too brittle to support the load, inducing its destruction in the friction track.

3.2.5 Coefficient of friction.

During the first 1000 cycles, the coefficient of friction is 0,65 with an oscillation on 1 cycle of ±0,15 (figure 7).

After 1000 cycles the coefficient of friction is stabilized at 0,6 and shown a lower oscillation, ±0,09.

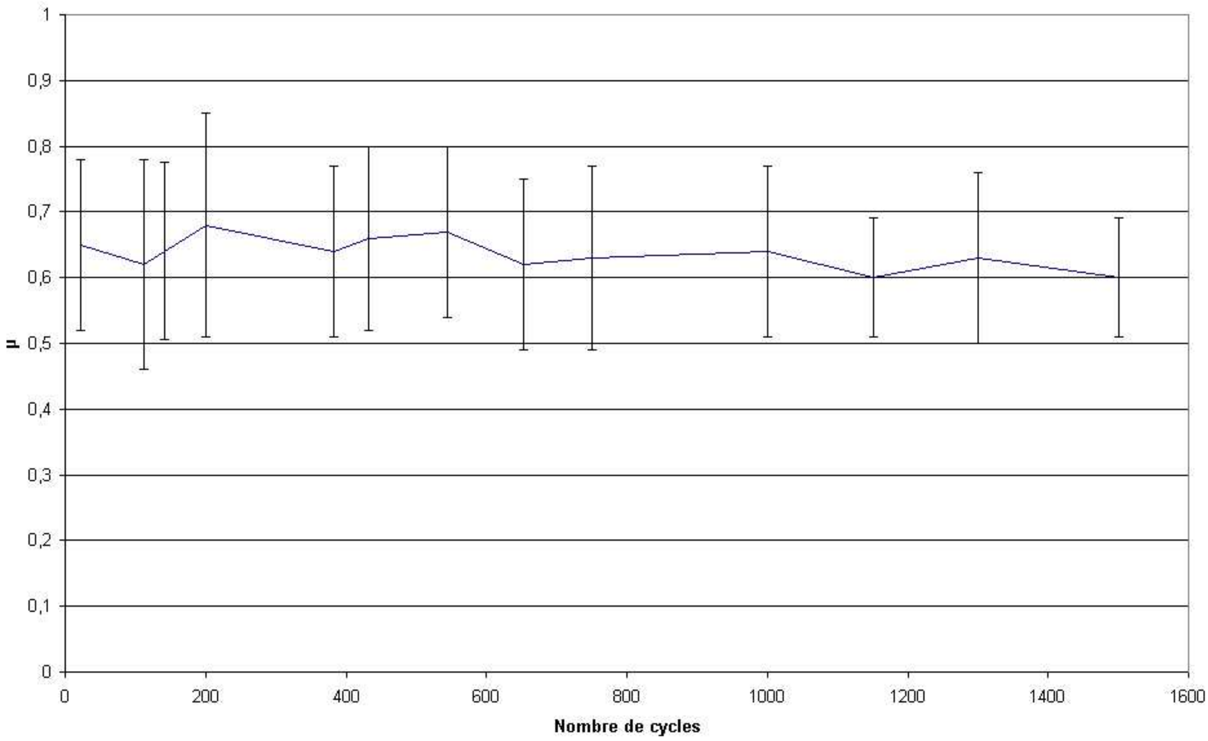


Figure 7 : Coefficient of friction of Zirconium during 1500 cycles under 800°C and 5 MPa of contact pressure.

3.2.6 Discussion.

The main result of this test is the excessive wear obtained with this coating, taking in account the performances seen in literature showing tests performed at 950°C and 5MPa of contact pressure or 800°C and 20MPa without excessive damages. The too low density of this coating could be one explanation of the low wear performance of this coating.

3.3 (ZrO_2-Y_2O_3 + CaF_2) / NiCrAlY / HR230 like on like.

3.3.1 Material description

To improve the tribological behavior of the previous zirconium coating, solid lubricant CaF_2 has been added to have a lower coefficient of friction and the density of the coating has been increased to limit the wear. The same Plasma process has been used, with the same coating methodology. The rate of CaF2 is : 75% vol. Zirconium Yttried TSP#204F*(tableau 3)* / 25% vol. CaF_2.

3.3.2 Experimental parameters.

The experimental parameters are :

- Temperature of 900℃,
- Contact pressure of 5MPa,
- Motion amplitude of 18 mm,
- Speed of 10 mm/s,
- Atmosphere : « pure He » (Tribometer feeding by Helium GC).

3.3.3 Working atmosphere.

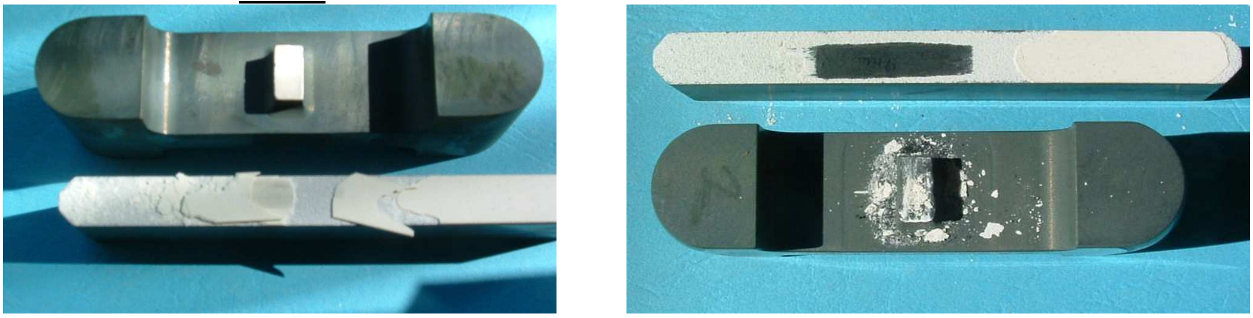
During the tests, the composition of the gas is well known and determined by sampling and chromatography analyses (table 5).

	H2 ppm	O2 ppm	N2 ppm	CO ppm	CO2 ppm
950℃ 1er dégazage	2392	<10	104	378	899
950℃ 2ème dégazage	2578	<10	115	2383	423
900℃ avant essai	nd	<10	25	51	24
900℃ après essai	nd	<10	28	90	31

Nd : No detected

Table 5 : Results of chromatography analyze after gas sampling.

3.3.4 Wear.



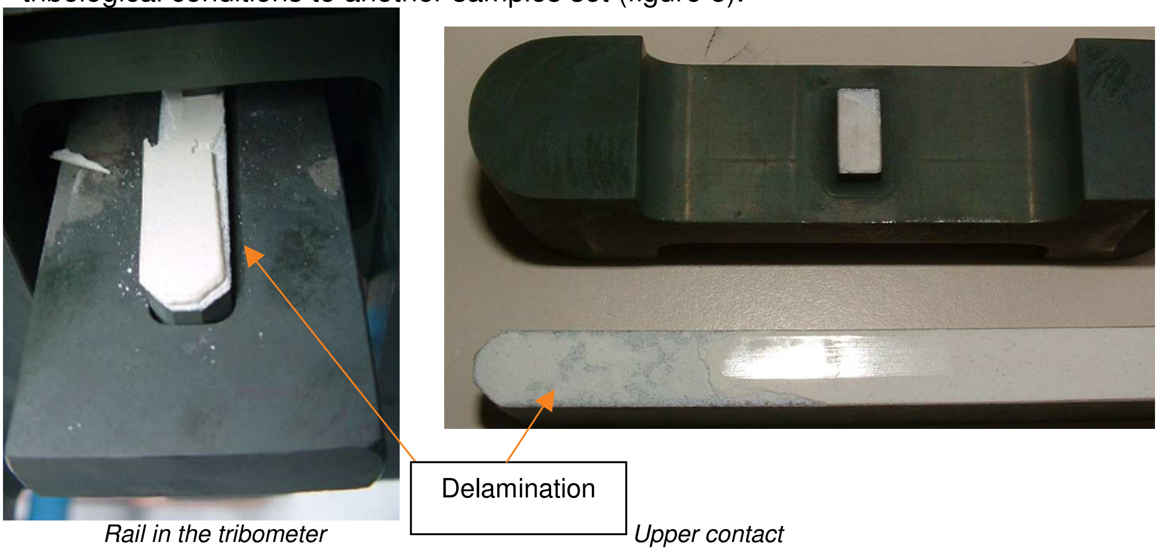
Upper Contact Lower Contact

Figure 7 : Sample of ZrO_2-Y_2O_3-CaF_2 / NiCrAlY after 719 cycles under 900°C and 5MPa of contact pressure.

Only 719 cycles have been made, before the shutdown of the tests induces by the fast increase of the coefficient of friction (see § 3.3.5).

The figure 7 illustrates the state of the samples after 719 cycles. The coating density and hardness are higher than the one without CaF_2, and the bond between the coating and the substrate is insufficient.

To verify, if this behavior is due to a defect on the coating of this sample, we applied the same tribological conditions to another samples set (figure 8).



Rail in the tribometer *Upper contact*

Figure 8: Sample of ZrO_2-Y_2O_3-CaF_2 / NiCrAlY after 1000 cycles under 900°C and 5MPa of contact pressure..

In this case, a delamination of the coating occurred, but the coating delaminated keep its place on the rail during friction. So no cue of this delamination could be seen with the evolution of the coefficient of friction.

3.3.5 Coefficient of friction.

The first test begun with a coefficient of friction of 0.4, which increased regularly during cycles, to hit an inhabitual value for this coating, with also very high perturbations of the coefficient of friction during one cycle (figure 9).

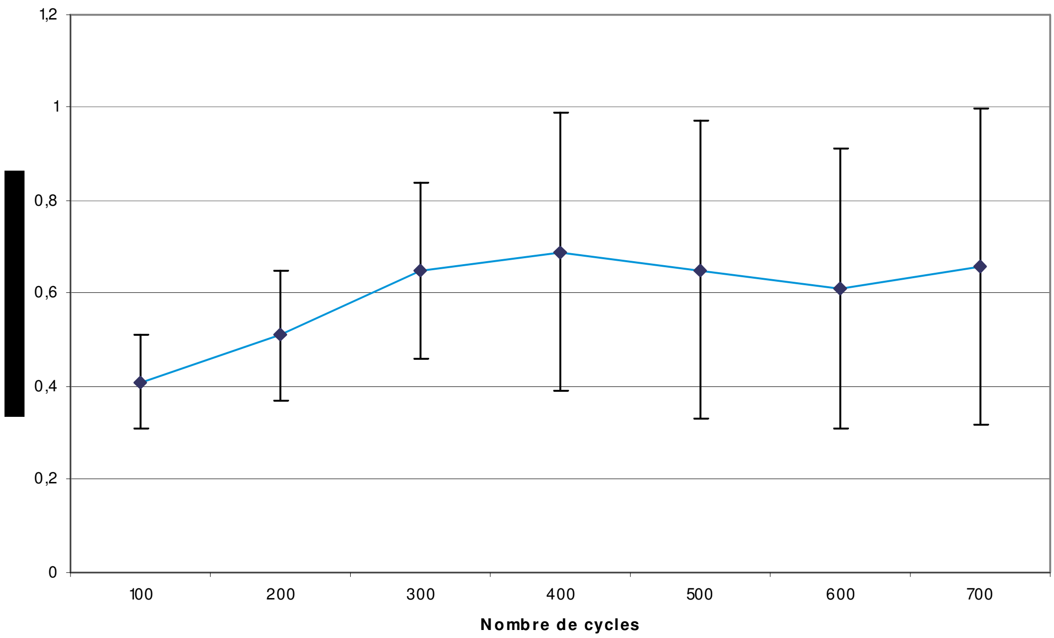


Figure 9 : coefficient of friction of the 1st sample of ZrO_2-Y_2O_3-CaF_2 / NiCrAlY during 719 cycles under 900°C and 5MPa of contact pressure

During the second test made in the same conditions, the coefficient of friction stayed low and stable all along the 1000 cycles (figure 10). As explained in the §3.3.4, the delaminated coating stayed in place, and the friction took place between the two coatings. This test highlight the real performance of the solid lubricant, CaF2, with a coefficient of friction of 0.27±0.025

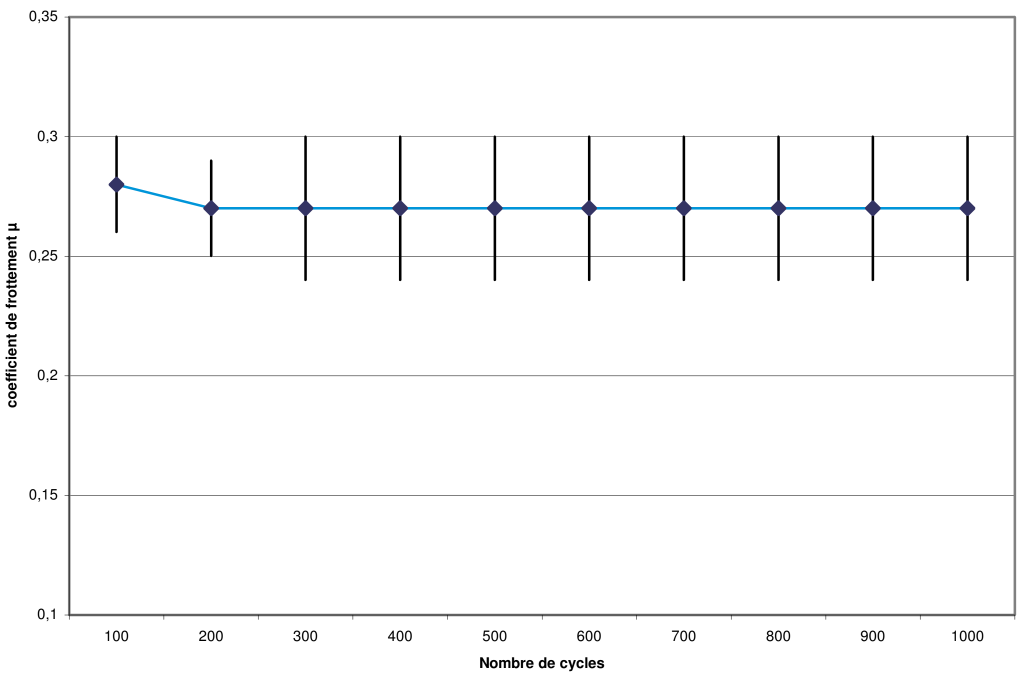


Figure 10 : coefficient of friction of the 2sd sample of ZrO_2-Y_2O_3-CaF_2 / NiCrAlY during 1000 cycles under 900°C and 5MPa of contact pressure

3.3.6 Discussion.

The program lead on the tribological efficiency of the add of CaF2 in Zirconium coating began with a test at 800°C and 5MPa of contact pressure (no contractual test in the WP5-HTR E), which alloyed us to compare it with the test made in the same conditions on the zirconium coating without solid lubricant. The Zirconium/CaF2 coating is more dense and harder than the Zirconium coating, and present a lower coefficient of friction and a better resistance to wear (5000 cycles without significative wear). The use of CaF_2 as a solid lubricant is very efficient.

The contractual tests show the limit of the accommodation of the NiCrAlY layer for temperatures higher than 800°C. With standard coating plasma process, the density of the coating has a major but opposed influence on its wear resistance and on its bond to the substrate :

- a mean density alloys a correct bond to the substrate but a bad wear resistance at low contact pressure at high temperature.
- Inversely, an high density alloys a high wear resistance but the coating became stiff and brittle and the bond with the substrate was embrittled.

So a trade of has to be made between wear resistance and the bond to the substrate to determine the optimum density of the coating.

The use of different coating process (like HVOF), and /or nanostructured powder could increase he coating density with increasing the bond to the substrate. It could be a way for future studies.

3.4 CERMET (Cr_3C_2 – NiCr) / HR230 like on like

3.4.1 Material description

CERMET Coatings are constitued of 75% Cr3C2 and 25% NiCr. They were obtained by spraying with HVOF (High Velocity Oxide Fuel) process. In this process, the hypersonic velocity of the flame, induce an high velocity to the powder, limiting the interaction time between the powder and the flame, decreasing the temperature of the powder compared to the other plasma process, and so, limiting the growth of the material grain. The material obtained are more dense and the final structure of the coating is near of the powder structure. The main characteristics of the coating are summarized in the table 6 and the process parameters in the table 7.

Revêtement HVOF de Carbure de Chrome liant NiCr (75/25)	
Référence : JK7184	Fournisseur : DELORO STELLITE

Description Générale		
Installation de projection	Type	HVOF
	Marque	DELORO
	Modèle	JET KOTE 2000
	Animation du pistolet	Robot ABB IRB 1400
Composition chimique du revêtement	Cr_3C_2	75%
	NiCr	25%
Granulométrie de la poudre	- 45 +15µm	
Caractéristiques du revêtement	micro-dureté	700 Hv0,3
	Macrodureté	89 Hrc
	Adhérence	> 70 MPa
	Porosités	< 2 %
	Epaisseur maximale	0,6 mm
	Température maximale de service	871 °C
	Rugosité brut de dépôt	Ra 5 à 8 µm

Tableau 6 : Coating characteristics.

Paramètres de Projection	
Gaz de combustion	O_2
	C_3H_6
Gaz de transport poudre	Ar
Pression O2	8 bars
Pression C3H6	5,5 bars
Pression Ar	7 bars
Débit O2	28 m^3 / h
Débit C3H6	16,5 m^3 / h
Débit Ar	0,9 m^3 / h
Débit de poudre	35 g / min
Buse	5/16 x 6
Injecteur	#50
Insert carbure	0.052"

Tableau 7 : Process parameters.

3.4.2 Experimental parameters.

The experimental parameters are :

- Temperature of 700°C,
- Contact pressure of 5MPa,
- Motion amplitude of 18 mm,
- Speed of 10 mm/s,
- Atmosphere : « pure He » (Tribometer feeding by Helium GC).

3.4.3 *Working atmosphere.*

During the tests, the composition of the gas is well known and determined by sampling and chromatography analyses (table 8).

Dates of sampling	Dates of chromat.	Cycles	Temperature	Pressure bar	O2 ppm vol	N2 ppm vol	CO ppm vol	CO2 ppm vol
26/05/04	11/06/04		100°C	2.23	16	89	11	134
			800°C after test	2.55	13	105	21	778
08/09/04	04/10/04	1000 to 2000 cycles	20°C	2.54	31	126	29	449
			700°C before test	2.53	151	616	25	686
			700°C after test	2.52	11	88	29	696
22/09/04	05/10/04	2000 to 3000 cycles	800°C dégazage	2.54	2034	7773	270	4422
			20°C	2.22	25	108	35	506
			700°C before test	2.56	29	108	23	956
			700°C after test	2.49	14	116	28	981
29/09/04	05/10/04	3000 to 4000 cycles	800°C dégazage	2.54	79	461	95	1725
			20°C	2.67	18	114	10	191
			700°C before test i	2.56	33	184	<10	377
			700°C after test	2.53	16	168	13	394
08/10/04	11/10/04	4000 to 5000 cycles	800°C dégazage	0.18	143	521	137	1760
			20°C	2.59	26	63	14	303
			700°C before test	2.58	<10	82	12	614
			700°C after test	2.45	<10	82	18	640

Table 8 : ***Result of chromatography analyze after gas sampling.***

3.4.4 Wear.

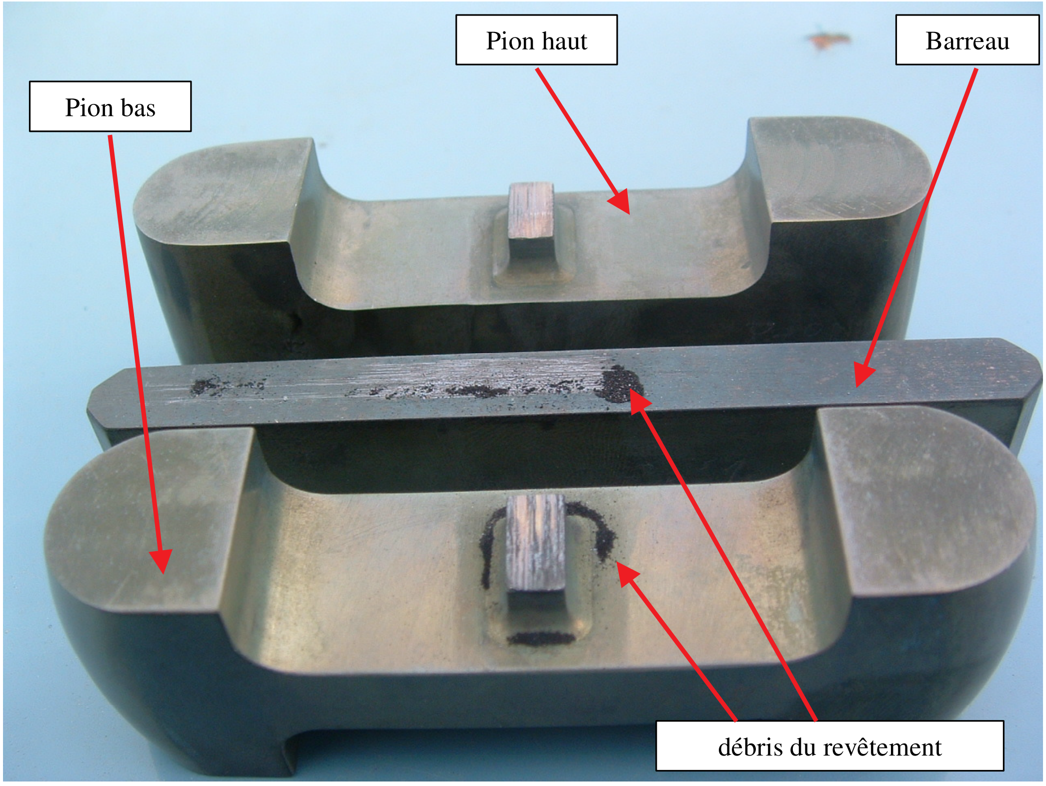


Figure 11 : CERMET samples after 5000 cycles under 700°C and 5MPa of contact pressure.

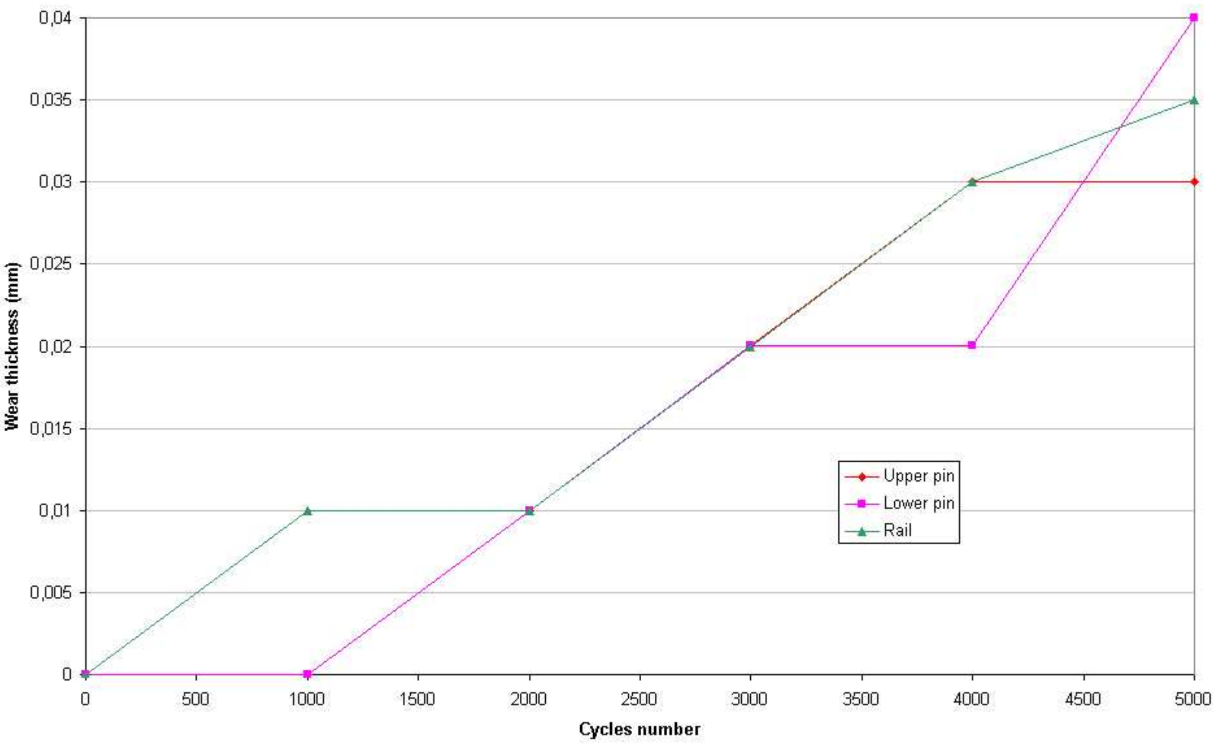


Figure 12: Wear of CERMET samples during the 5000 cycles under 700°C and 5MPa of contact pressure.

The presence of debris like powder (see 11 after samples removal), show a tribological accommodation made essentially by abrasion.

The metrologies made on the samples each 1000 cycles (figure 12), show a low wear with a mean value of 0.008 µm/cycle.

3.4.5 Coefficient of friction.

During the 5000 cycles, the mean coefficient of friction is 0,28±0,03 (figure 13), low value for a no lubricate contact.

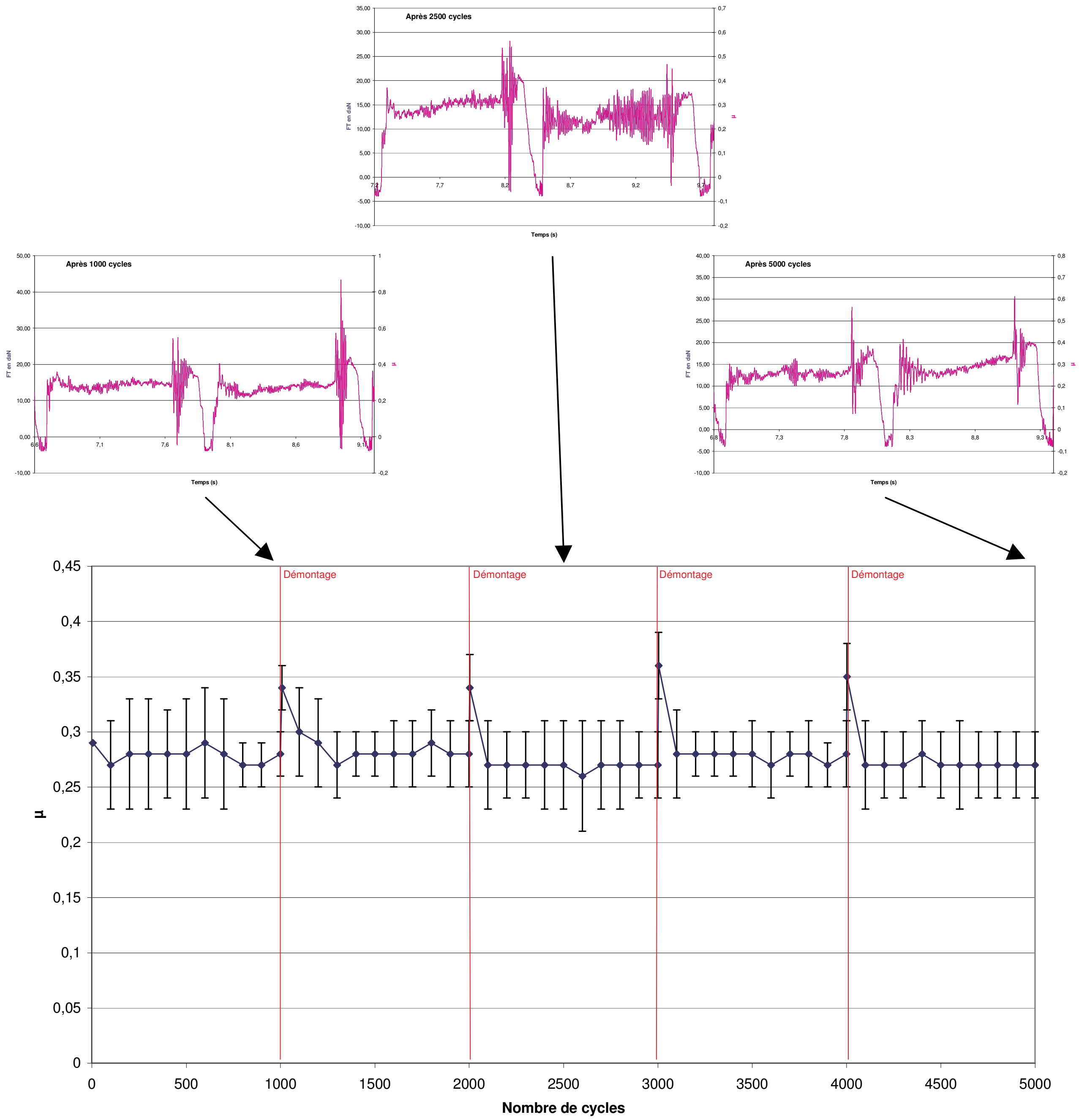


Figure 13 : Coefficient of friction during 5000 cycles under 700°C and 5MPa of contact pressure.

The coefficient of friction has a stable mean value. Its fluctuation is higher during the first 1000 cycles, when the accommodation of the surfaces is the highest. After each samples removal, the contact is drained of its debris. This induces a temporary increase of the coefficient of friction, for the next cycles, up to the re-generation of the debris layer in the contact (figure 13).

3.4.6 Discussion.

The CERMET (Cr3C2 – NiCr) has a good tribological behavior at 700 °C and 5MPa of contact pressure.

3.5 SiC / HR230 like on like

3.5.1 Material description

SiC is a particular material to be coated. In a first time it is planed to made thick coating by plasma processes. No company was found to made this process. The technical issue comes from the material physical characteristics of SiC which has sublimation tendency at high temperature.

SiC coatings are finally obtained by PVD processes. It is a pulverization of a pure Si target, in a reactive plasma, made of Argon and Acetylene. It is a critical proportion of acetylene which allows the SiC generation. The thickness of the coating is about 5µm.

Figure 14 : SiC after PVD coating

After SiC PVD coating, samples present refringence rays on the coated surfaces, showing a gradual structure difference of the coating.

3.5.2 Experimental parameters.

The experimental parameters are :

- Temperature of 900°C,

- Contact pressure of 5MPa,
- Motion amplitude of 18 mm,
- Speed of 10 mm/s,
- Atmosphere : « pure He » (Tribometer feeding by Helium GC).

3.5.3 Working atmosphere.

During the tests, the composition of the gas is well known and determined by sampling and chromatography analyses (table 9).

		For 3 bar				
Cycles		H_2 ppm	O_2 ppm	N_2 ppm	CO ppm	CO_2 ppm
0 to 1000	1st disgasing	2820	87	2867	1097	1413
	2sd disgasing	nd	11	42	15	98
	900°C before test	nd	<10	39	<10	70
	900°C after test	nd	<10	43	18	96

Table 9 : Result of chromatography analyze after gas sampling.

3.5.4 Wear.

After removal the samples, the observation of the surfaces of friction shown the disparition of the coating on the wear track (figure 15). The coating was to thin and to brittle to run under such conditions : 5 MPa and 900°C.

Upper Contact — Lower Contact

Figure 15 : Sample of SiC after 1000 cycles under 900°C and 5MPa of contact pressure.

In the wear track, the maximum depth is about 0,1 to 0,15 mm after 1000 cycles.

Taking into account the results obtained on tribological tests preformed on Hr230 like on like contact at 450°C and 5MPa of contact pressure (§3.1.), the wear is not so excessive. To explain this, 2 assumptions :

- The debris, issued of the coating crack, limit the contact of the 1st body made of HR230 and so limit the adhesive component of the wear,
- and the wear due to the abrasion with the hard SiC debris is smooth (like lapping).

3.5.5 Coefficient of friction.

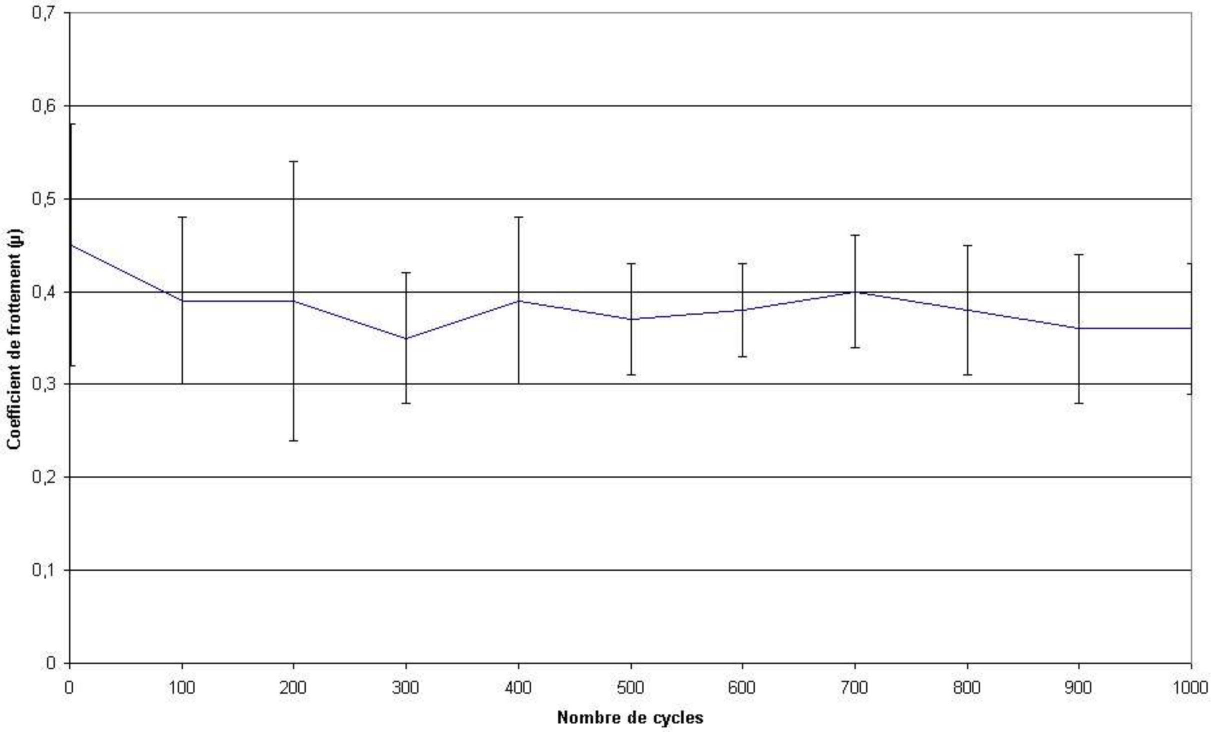


Figure 16 : Coefficient of friction of SiC coating during 1000 cycles *under 900°C and 5MPa of contact pressure.*

The mean coefficient of friction is 0,38±0.08 lower and less variable than with an HR230 like on like contact. This is a confirmation of the minimization of the adhesive component in the contact behavior.

3.5.6 Discussion.

The coating is too thin and to brittle, and the substrate too soft at 900°C to support the load of 5 MPa. This induce the failure of the coating.

SiC coating made by PVD process is not adapted to run in these conditions.

4. CONCLUSION.

Within the framework of the European Program HTR-E of the WP5, this document puts the emphasis on the CEA contribution of the deliverable D41 which aims to carry out a set of elementary tests in helium environment for CGR applications. The main conclusions for each test are (table 10) :

- **HR230 :** these tests were performed at 450°C and 5 MPa of contact pressure. The results are foreseen for a metallic contact at high temperature : the main tribological mechanism is adhesion.

- **CERMET** (Cr_3C_2 – NiCr) : The test were performed at 700°C and 5 MPa of contact pressure. This is the single test, in this program, in which the 5000 required cycles were performed. The tribological behavior is very good, with a wear rate of 0.008 µm/cycle and a coefficient of friction lower than 0.3.

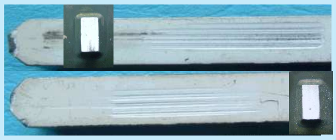
- **Zirconium coating**s :

  - ***ZrO2-Y2O3*** */* ***NiCrAlY*** (Classic plasma spraying) : the test was performed at 800°C and 5 MPa of contact pressure. Only 1600 cycles were performed. The wear and the coefficient of friction (µ=0.6) are too high.

  - ***75% (ZrO2-Y2O3) + 25% CaF2*** */* ***NiCrAlY*** (Classic plasma spraying) : the test was performed at 900°C and 5 MPa of contact pressure. The wear and the coefficient of friction (µ=0.3) were low, but thermal expansion between the coating and the substrate induced delamination of the coating.

To improve the tribological behavior of zirconium coating the use of solid lubricant like CaF2 is very efficient. The improvement of both the bond between the coating and the substrate and the density, in order to reduce the wear, could be obtained by the use of more energetic spraying processes like HVOF or D-gun.

- **SiC** (PVD) : the test was performed at 900°C and 5 MPa of contact pressure. The coating is too thin and too brittle, and the substrate too soft at 900°C to support the load of 5 MPa. This induces the failure of the coating.

Coating	Temperature (°C)	Contact pressure (MPa)	Coefficient of friction	Nb of cycles	Wear	
HR 230	450	5	0,4±0,15	2600	-	
ZrO_2-Y_2O_3 (PLASMA SPRAYING)	800	5	0,65±0,1	1500	-	
[ZrO_2-Y_2O_3] + 25%CaF_2 (PLASMA SPRAYING)	900	5	0.4 to 0.8	716	*Coating delamination*	
	900	5	0,25±0.05	1000	+ *but Coating delamination*	
CR_3C_2 – NiCr (HVOF)	700	5	0,28±0,03	5000	++ *0,008µm/cycle*	
SIC (PVD)	900°C	5	0,38±0.08	1000	---	

- - : total coating degradation , - - : big wear, - : wear, 0 : mean wear,
+ : low wear, ++ very low wear.

Table 10 : Synthesis of tribological results obtained in the frame of HTRE – WP5 European program.

5. APPENDICES

APPENDICES

5.1 Appendix 1 : NNC Comments

NNC Comments on the CEA tests in D41

Some general points:

All the wear mechanisms can be classified as adhesive although there is clearly a significant contribution from ploughing. This is always a problem with a flat-on-flat geometry. The erratic nature of the friction traces, the increase in friction during a cycle and the non-uniform wear along the wear track all seem to indicate some kind of misalignment or uneven loading may be taking place. A large radius pin would be a good alternative but some effort should be made to produce more even wear and friction.

In general, wear depths ought to be measured after a test by profilometry. The wear traces do not give maximum wear depth so will not necessarily indicate when a coating has been penetrated. Wear rates quoted in µm per cycle cannot be used to compare the results with others found elsewhere. The wear rate could be calculated from the wear profile and quoted in m^3/Nm. A value of $\sim 10^{-14}$ m^3/Nm would indicate a mild (low) wear rate. Even without profilometry an estimate could be made of wear rate in m^3/Nm.

Care should be taken not to refer to load when it is a pressure e.g. 5 MPa.

It is incorrect to say that a coating is too brittle to support the load. Better to say the coating is too brittle to resist the shear forces.

Some specific points:

It is not clear why the HR230 vs HR230 test were carried out at 450℃ and not 500℃. This test gave rise to severe adhesive wear (galling). I think the friction would be better described as erratic, with a coefficient of up to 0.8. I think it would be best to say that this combination behaved poorly, as expected.

There is no wear data for the ZrO_2 test. We would therefore not rule out ZrO_2 on the basis of this test.

Ploughing had a disastrous effect on the ZrO_2/CaF_2 coating.

How thick is the Cr_3C_2–NiCr coating? Why was the test at 700℃ and not 750℃? The results do look promising, as expected on the basis of the UK programme, but unfortunately have been spoiled to some extent by the equipment problem.

Yes, a 5 µm SiC coating is not suitable on HR230. An additional undercoat might help.

Comments on specific tests

HR230, like-on-like - 450℃, CEA

As expected, the tests on HR230 vs HR230 at 450℃ gave rise to severe adhesive wear (galling), with a friction coefficient of up to 0.8. In a like-on-like configuration, HR230 is clearly unsuitable for rubbing applications at 450℃, and most probably at all temperatures in the range of interest.

HR230 + NiCrAlY + ZrO_2-Y_2O_3, like-on-like - 800℃, CEA

The poor result produced by this preliminary test, at 800℃, was unexpected and should not be used to discount this coating from application. The intrinsically high friction coefficient of the coating will preclude its use in some situations.

HR230 + NiCrAlY + ZrO_2-Y_2O_3/CaF_2, like-on-like - 900°C, CEA

In the 900°C test, the coating delaminated. More testing is required on this system of intrinsically low friction coefficient, which shows promise for low load situations.

HR230 + Cr_3C_2/Ni-Cr, like-on-like - 700°C, CEA

This coating has found wide applicability in helium and carbon dioxide-cooled systems in the UK. In the CEA test, the coating was shown to wear moderately and displayed a low friction coefficient (mean of 0.28). This good result confirms the suitability of this coating for use in sliding situations at high temperature in helium.

HR230 + SiC, like-on-like - 900°C, CEA

In this test on a PVD SiC coating, heavy wear occurred. The coating apparently wore through so early in the test that no sensible frictional information was obtained. The thin coating clearly needs a supportive undercoat. A 100 µm NiCrAlY coating could be used as for the ZrO_2-Y_2O_3 coating. The results of further tests are required before an assessment could be made of the applicability of this coating system.

Conclusions

The preliminary tests have given some useful indications but in order to obtain some results in which we can have confidence the equipment should be re-examined and the misalignment or uneven loading rectified for the FP6 tests. We think it is essential to consider going to a sphere-on-flat configuration.

5.2 Appendix 2 : CEA answer on NNC Comments

Misalignement on CEA tribometer

The design of this tribometer has been made to avoid **misalignement** : it is the load and the samples surfaces which align the samples by means of spherical …and there is no static indeterminacy in sample guidance.

I agree that plane / plane contact is the most difficult situation in tribology, but it is also the most realistic for an industrial process and it is in that way this tribometer was designed. The "erratic nature of friction traces" are classical in plane / plane contact because of 1st (and 2sd) order surface defects. When the evolution of the contact goes to an improvement of the surface aspect, the "erratic nature of friction traces" disappears after the lapping phase.

Wear depths measurement

I agree that profilometry gives better accuracy on wear depths measurement. Nevertheless, for thick coatings, with low wear rate, our methodology allowed to estimate wear rate and to see if the coating was penetrated. After that, SEM observations is a very useful and determining tool to validate this last point.

Concerning the criteria to evaluate the wear, I think that you know that each tribometer has its signature on the results it provides. So even if I used the criteria you mentioned, it would be hazardous to compare it with those found on a pin on disc tribometer. In a plane / plane contact, the wear debris trapped in the contact constitute the third body, which is different in the case of sphere / plane contact. If the 2 first bodies produce a protective third body, (it is the case with CERMET), and if the contact has the capability to keep it inside, (it is also the case with plane / plane contact), it is a protection against the wear. Pin on disc tribometer and CEA tribometer can't give the same results for same tribological conditions (except contact).

SiC coating is too brittle

I don't think it is incorrect to say that SiC coating is too brittle to withstand the load, and I don't think that is the shear loads that break the coating. In fact this is a combination of 2 phenomenons :

- SiC coating is very hard and brittle and so not very resistant to high deformations (flexion),
- HR230 substrate, at 900°C, has a decrease of its stiffness,

So under 5 MPa of contact load, the deformation of the substrate is too large and the SiC is too brittle to accommodate.

I think that if SiC would have been coated on a surface more rigid, i.e. on a CERMET or NiCrAlY coating, its tribological behavior would be different. This phenomenon has been highlighted on others CEA tribological tests.

Ploughing had a disastrous effect on the ZrO_2/CaF_2 coating

Ploughing is the consequence. The issue here is the delamination of the coating due the too high differential thermal expansion between the coating and the substrate. After delamination, the coating trapped in the contact breaks itself in hard debris which induce ploughing of the substrate.

Unfortunately have been spoiled to some extent by the equipment problem

I maintain that there is no equipment problem ? Several other tests performed with CERMET coating, and not only the one reported in the D41, were quite conclusive and show that CERMET is an excellent coating with performances higher than those of the European contractual test. And in these cases, they have not been spoiled at all. .

Gap of temperature for tests in HR230 and CERMET.

In the first tests the thermocouple taken to regulate the temperature of the furnace have a gap of 50°C with the temperature of the samples. For the other tests changes have been made to regulate on the samples temperature.

The others comments are in agreement with what I wrote in the D41.