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Tribology: a compendium of recommendations

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ABSTRACT

In the frame of HTR-E work package 5 about tribology, the main tasks are as follow:

- Task 1: state of the art
 - ♦ Task 1.1: Review of tribological problems in former HTRs and FBRs
 - ♦ Task 1.2: Tribological conditions in PBMR and GTMHR
 - ♦ Task 1.3: Review of existing materials and coatings
- Task 2: Proposal of elementary tests
- Task 3: Tests in Helium conditions
 - ♦ Task 3.1: Realization of elementary tests
 - ♦ Task 3.2: Tribology: a compendium of recommendations

The main objectives of this document are:

- to summarize the state-of-the-art made in task 1;
- to describe the tests and involved equipment;
- to give the interpretation of the test results for the different proposed materials.

A compendium of recommendations defining particularly the remaining uncertainties is also summarised in this report.

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Appendix 2 DTN/STPA/LTCG/2006-02	Tribology: a compendium of recommendations – D42

1. REFERENCES

- | | |
|-----------------------------|---|
| [1] HTR-E-02/03-D-5-0-1 | D36 – Work plan |
| [2] HTR-E-02/11-D-5-1-1 | D37 – Review of tribological problems on former HTRs and on FBR |
| [3] HTR-E-02/11-D-5-1-2 | D38 – Review and identification of typical HTR tribology conditions of components |
| [4] HTR-E-03/06-D-5-1-3 | D39 – Review of existing materials and coatings |
| [5] HTR-E-03/11-D-5-2-1 | D40 – Proposal of elementary tests |
| [6] HTR-E-05/12-D-5-3-1-1/2 | D41 a – Realisation of elementary tests (CEA) |
| [7] HTR-E-05/12-D-5-3-1-2/2 | D41 b – Realisation of elementary tests (FANP) |

2. INTRODUCTION

In the frame of HTR-E work package 5 about tribology, the main tasks are as follow:

- Task 1: state of the art
 - ♦ Task 1.1: Review of tribological problems in former HTRs and FBRs (D37)
 - ♦ Task 1.2: Tribological conditions in PBMR and GTMHR (D38)
 - ♦ Task 1.3: Review of existing materials and coatings (D39)
- Task 2: Proposal of elementary tests (D40)
- Task 3: Tests in Helium conditions
 - ♦ Task 3.1: Realization of elementary tests (D41)
 - ♦ Task 3.2: Tribology: a compendium of recommendations (D42)

The main objectives of this document are:

- to summarize the state-of-the-art made in task 1;
- to describe the tests and involved equipment;
- to give the interpretation of the test results for the different proposed materials.

A compendium of recommendations defining particularly the remaining uncertainties is also summarised in this report.

3. STATE OF THE ART

The state of the art is summarized in documents [2] to [4].

The main feedbacks extracted from past experiences gained in different countries are as follows:

3.1. From document [2]

- From Dragon experience (UK): wear and adhesion in higher temperature areas: need of coatings;
- From AGR experience (UK): Fretting was considered to be an important issue, also wear and galling;
- HTR Module test (G): friction tests, static welding tests, aging tests in dry HTR helium at temperatures up to 1000°C;
- Fort St Vrain (USA): high moisture resulting in corrosion of stainless steel control rods cables;
- HTTR (J): coating of the hot gas duct with 3 µm TiN to prevent from seizure and excessive wear of the liner slides (Hastelloy surfaces) of the auxiliary concentric hot gas duct for the HTTR at a 950°C;
- HTR10 (Ch): requires special lubrication for control rod system components which have to operate at high temperatures and high radiation fields.

3.2. From document [3]

- Identified types of degradation: static adhesion, wear, fretting, rubbing, erosion
- Temperatures: from <150°C to 900°C
- Components: valves, bearings, plate supports (recuperator), drive mechanism (CRDM), core elements, hot gas duct, ...
- Establishment of filling cards for GTMHR with:
 - ♦ General information
 - ♦ Parts in contact dimensions
 - ♦ Tribological conditions
 - ♦ Environment conditions
 - ♦ Observations

3.3. From document [4]

- Data from: Materials & Coatings in UK AGR and in Dragon, HTR Module (G), Turbines, Aerospace
- 100-500°C: Cermet Cr_3C_2 / NiCr, Ni based alloy, solid lubricants, WS, cubic boron nitride (BN), TiN, Zirconia / NiCrAlY, ...
- 500-800°C: Nickel alloys (650°C), Cermet Cr_3C_2 / NiCr, cermet $\text{WC}_3/\text{Cr}_3\text{C}_2/\text{NiCr}$, Zirconia / NiCrAlY, solid lubricants, ...
- 800-1000°C: Cermet, Zirconia / NiCrAlY or Alumina based coating, TiC(Ni₃Ti) and NbC(NiCr), solid lubricants, ...

4. TEST DESCRIPTION AND INVOLVED EQUIPMENT

Two devices have been used for tribological experimentation in GCR condition:

- CEA, He Tribometer;
- FRAMATOME, Pin/disc device.

Each tribometer covers two main domains, the first one for classical tests (FANP) and the second one devoted to high temperature, and low pressure (CEA). The Figure 1 presents both domain covered by each tribometer.

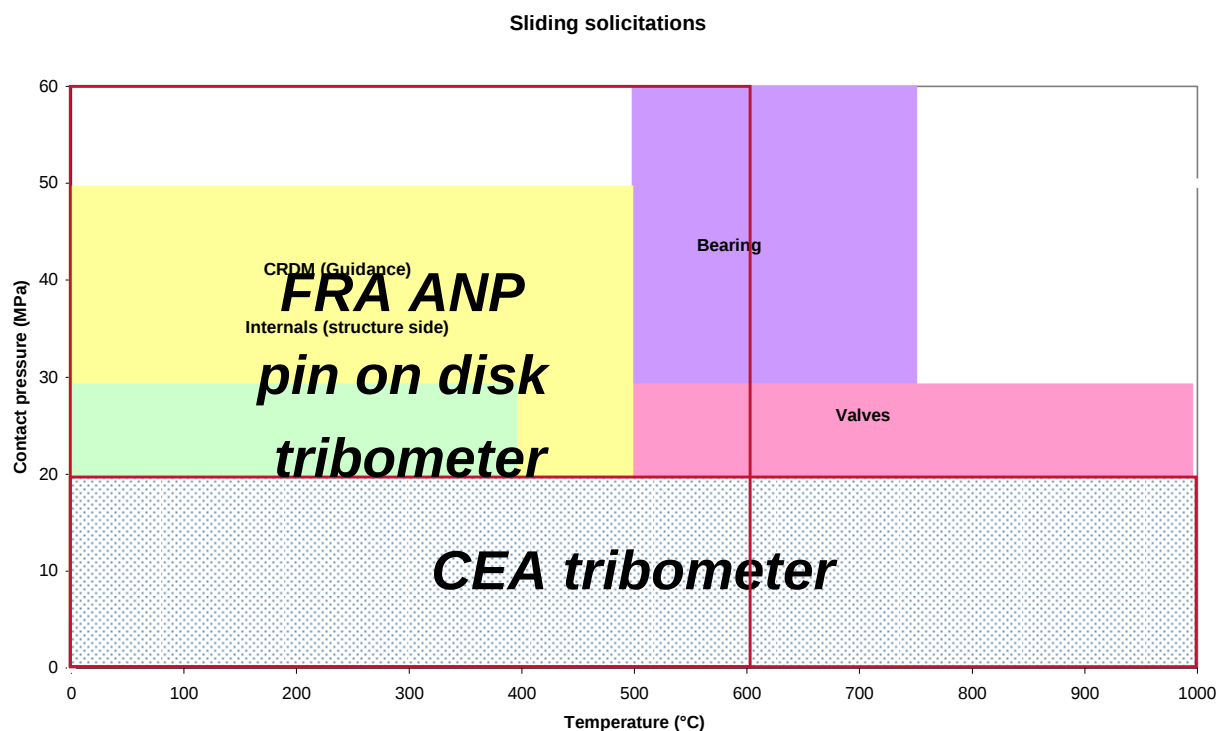


Figure 1: tribometers domains and sliding sollicitation

4.1. CEA – He Tribometer

“He tribometer” is a pins on rail alternative sliding tribometer. This apparatus is made in two parts (Figure 2):

- 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.

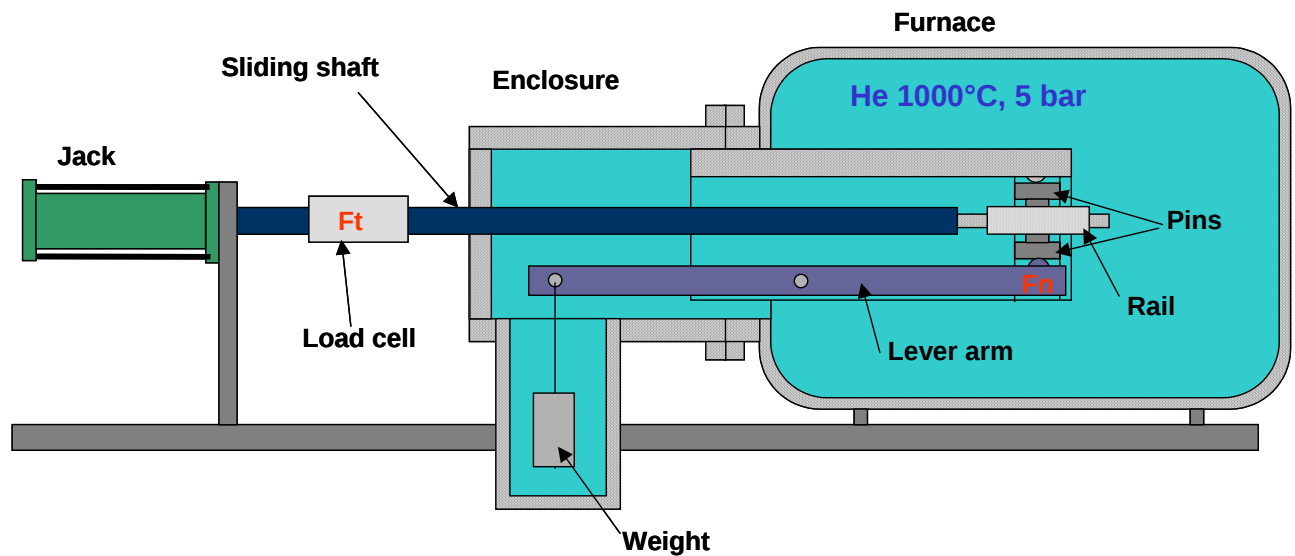


Figure 2: schematic diagram of CEA He tribometer.

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,
 - ♦ Steam, impurities

4.2. FRAMATOME ANP - Pin/disc device

General features (Figure 3):

- Fixed pin and rotating disk;
- Thermal housing around the disk;
- Normal load generated by free mass or spring;
- Little capacity for test in humid or lubricated environment;
- Contact forces (normal and tangential) measured by strain gauge sensors.

Performances:

- Normal load up to 2,5 daN (estimated contact pressure: from 2 MPa to 600 MPa);
- Sliding speed: 1 to 250 mm/s;
- Temperature up to 600°C;
- Working environment: liquid (at low temperature) or gas;

- Nature of contact: typical sphere on plate or piece of real components;
- Measurement of temperature, displacement (wear evolution), normal and tangential forces, distance covered with personal computer during test.

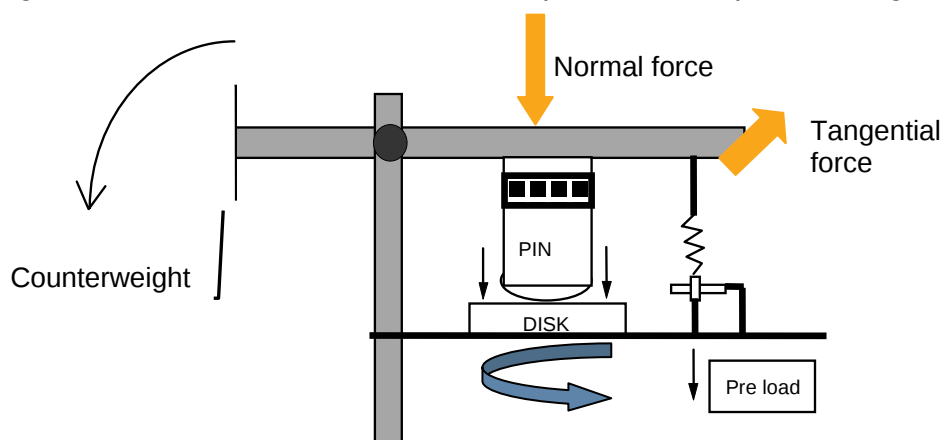


Figure 3: FRAMATOME ANP pin-on-disk tribometer

5. INTERPRETATION OF THE TEST RESULTS FOR THE DIFFERENT PROPOSED MATERIALS

5.1. performed tests

All tests have been realized in pure Helium, with several coatings and 2 substrates (see Table 1).

N°	Component / contact	Material 1	Material 2	Temper. (°C)	Load (MPa)	Covered length (m)	Tribometer FANP	Tribometer CEA
2	Reference test	HR230	HR230	450	5	100		
4	Liner / Liner	HR230 + ZrO ₂ /Y ₂ O ₃	HR230 + ZrO ₂ /Y ₂ O ₃	450	5	200		
6		HR230 + ZrO ₂ /Y ₂ O ₃	HR230 + ZrO ₂ /Y ₂ O ₃	800	5	200		
8		HR230 + ZrO ₂ /Y ₂ O ₃ /CaF ₂	HR230 + ZrO ₂ /Y ₂ O ₃ /CaF ₂	450	5	200		
10		HR230 + ZrO ₂ /Y ₂ O ₃ /CaF ₂	HR230 + ZrO ₂ /Y ₂ O ₃ /CaF ₂	900	5	200		
14	Liner / Liner	HR230 + SiC	HR230 + SiC	900	5	200		
18		HR230 + Cr ₃ C ₂	HR230 + Cr ₃ C ₂	700	5	200		
20	Internals / vessel or Internals / block support	9%Cr	9%Cr	250	10	200		
22		9%Cr	"	450	10	200		
24		9%Cr + TiC	9%Cr	250	10	200		
26		9%Cr + TiC	"	450	10	200		
28		9%Cr + WC/NiCr	9%Cr	250	10	200		

Table 1: Realized tests

5.2. Test results

All results are given in documents [6] and [7].

The relevant test results can be summarized as follows:

- The best results have been obtained at 700°C on CERMET coating (Cr_3C_2 -NiCr) deposited by HVOF;
- The Coating Zirconia ($\text{ZrO}_2/\text{Y}_2\text{O}_3$) + solid lubricant (CaF_2) has to be improved (adhesion) for a resistance at 900°C;
- SDG2005 (WC, WC_3 / Cr_3C_2 , Ni) deposited by Super Detonation Gun has a good behaviour at 250°C (but attention has to be paid to the material in contact)

6. RECOMMENDATION COMPENDIUM

This paragraph deals with recommendation compendium, including the remaining uncertainties.

The main recommendations are:

- Necessity to elaborate a technical specification for coating manufacturing (coating thickness in regard of solicitation, bonding with substrate, characterization methods, thermal expansion difference effects, ...);
- Impurity content in helium shall be specified (dedicated tribometer with an adapted gas injection);
- Need of regulation about tribological tests. There is a clear need to produce results under standard conditions and the preliminary tests have provided such information. In further test work, it will be necessary to expand the standard conditions to include a small number of different loadings for different coatings, to address the conditions of their application. Also, fretting wear must be addressed in reciprocating sliding tests at high frequency;
- The testing has confirmed the good wear resistance of $\text{Cr}_3\text{C}_2/\text{Ni-Cr}$ and WC/Ni-Cr coatings in a helium atmosphere, in their temperature ranges of applicability. $\text{ZrO}_2\text{-Y}_2\text{O}_3$, with and without an incorporated solid lubricant, shows potential for PBMR and GTMHR applications. Other, thin, coatings may have some applicability but will not find wide application. Further work must identify a small number of coatings that can provide wear resistance, and the required frictional properties, for components at all temperatures of interest. Those coatings identified to date will meet many of the requirements;
- In the sliding tests carried out so far, adhesive wear has dominated, as would be expected. Some coatings have failed through lack of support and others through ploughing, especially in the flat-on-flat configuration. Although such a configuration is desirable in reproducing some conditions on plant, it brings experimental difficulties. For close correlation in the results from the two laboratories, it may be necessary to adopt a large radius-on-flat configuration as standard. Except in the very early stages of a test when little wear will have occurred, this configuration will be essentially flat-on-flat.
- Tests have been realized with a sliding movement. In the future, there is a need to test other types of movement (sliding, dwell motion, fretting, ...);

- Zirconium and CERMET coatings seem to be the best candidates for high temperature helium conditions (coating adhesion at high temperature should be improved);
- For temperatures lower than 500°C, there is no real issues to find a couple of material or coating which is suitable for tribology application in gas cooled reactor conditions;
- Metal like on like tribological contact induce excessive wear by adhesive mechanisms;
- Concerning thin coatings, like TiN or SiC, tribological tests confirmed that they are not adapted at GCR conditions;

The test methods and the feedback of test results will be useful for RAPHAEL IP work package 3 of SP-CT sub-project.

Appendix 1

C6940/TR/007: NNC contribution to HTR-E WP5 Deliverable D42 (Analysis of the application of the results on PBMR design: recommendations)

**NNC contribution to HTR-E WP5 Deliverable D42
(Analysis of the application of the results on PBMR
design: recommendations)**

DOCUMENT ISSUE RECORD

(engineering documents)



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1.0 INTRODUCTION

Potential tribological problem areas in PBMR were identified in D38 on the basis of a review of UK operational experience with helium and CO₂-cooled reactor systems. The review addressed tribological concerns in the various parts of the system. In the core, the main concerns are graphite-graphite and graphite- steel rubbing contacts under the action of gas flows and temperature changes. For the control and fuelling system, tribological concerns include static adhesion and adhesive wear. The reactor cooling system includes heat exchangers, in which adhesive wear and fretting, under the action of thermal expansion and vibration, are the principal concerns. Fretting is also a concern in the blowers. In the power conversion unit, which includes high and low pressure turbo units, fretting and adhesive wear are again concerns, as is blade erosion. In the ancillary equipment, including compressors and valves, adhesive wear and static adhesion are concerns. Temperatures of interest range from 450–900°C and above in the core, 700°C to 950°C in the turbines, from 500-700°C or more in the recuperator and heat exchangers down to 150°C in parts of the heat exchangers and core conditioning system.

Materials envisaged for PBMR application include austenitic stainless steels, nickel-based alloys, chromium steels and low alloy steels. To date, tests have been carried out on the nickel-based superalloy HR230 and the 9% chromium steel Z10CDVNB91, uncoated and coated. Several coatings have been tested, including the cermet Cr₃C₂/Ni-Cr that has been widely applied in UK gas-cooled systems. The following comments (section 2) are on the tests carried out in pure helium by Framatome and CEA. The Framatome tests were carried out on a pin-on-disc (continuous sliding) tribometer; the contact was sphere-on-flat. The CEA tests were carried out on a pin-on-plate (reciprocating sliding) tribometer; the contact was flat-on-flat.

2.0 COMMENTS ON THE TEST RESULTS

2.1 HR230, like-on-like

450°C, Framatome 450°C, CEA

As expected, the tests on HR230 vs HR230 at 450°C 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°C, and most probably at all temperatures in the range of interest.

2.2 HR230 + NiCrAlY + ZrO₂-Y₂O₃, like-on-like

450°C, Framatome 800°C, CEA

HR230 with a coating of yttria-stabilised zirconia on an NiCrAlY undercoat was shown to wear moderately in the Framatome test in a like-on-like configuration at 450°C, with a wear rate of $\sim 3 \times 10^{-13} \text{ m}^3/\text{Nm}$. The friction coefficient remained high, at ~ 0.8 , throughout the test. At 800°C, the coating wore badly, the friction coefficient oscillating between 0.5 and 0.8 during each cycle of sliding contact. The poor result produced by this preliminary test, at 800°C, was unexpected and should not be used to discount this coating from PBMR application. The intrinsically high friction coefficient of the coating will preclude its use in some situations.

2.3 HR230 + NiCrAlY + ZrO₂-Y₂O₃/CaF₂, like-on-like

450°C, Framatome 900°C, CEA

With the solid lubricant CaF₂ incorporated into the ZrO₂-Y₂O₃ coating, wear was greatly reduced at 450°C. During the test, the solid lubricant was gradually removed from the wear track on the disc, the friction coefficient building from 0.35 to 0.7. 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.

2.4 HR230 + Cr₃C₂/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.

2.5 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 ZrO₂-Y₂O₃ coating. The results of further tests are required before an assessment could be made of the applicability of this coating system for use in PBMR.

2.6 Z10CDVNB91, like-on-like

250°C, Framatome 450°C, Framatome

This combination wore severely at both temperatures and gave rise to high values of friction coefficient. The Z10CDVNB91 alloy clearly has no potential for use in rubbing situations without a coating.

2.7 Z10CDVNB91 vs Z10CDVNB91 + WC/Ni-Cr

250°C, Framatome

In this test, the uncoated pin wore severely, while the coated disc remained hardly worn. As expected the WC/Ni-Cr coating has provided excellent wear resistance at 250°C. There is now a clear need to perform tests in a like-on-like configuration at temperatures up to 450°C.

2.8 Z10CDVNB91 vs Z10CDVNB91 + TiC

250°C, Framatome 450°C, Framatome

Again, a test on a very thin coating has produced early penetration. Like SiC, this coating requires a hard undercoat, capable of supporting the thin coating under the test load.

3.0 CONCLUDING REMARKS

The testing has confirmed the good wear resistance of $\text{Cr}_3\text{C}_2/\text{Ni-Cr}$ and $\text{WC}/\text{Ni-Cr}$ coatings in a helium atmosphere, in their temperature ranges of applicability. $\text{ZrO}_2\text{-Y}_2\text{O}_3$, with and without an incorporated solid lubricant, shows potential for PBMR application. Other, thin, coatings may have some applicability but will not find wide application. Further work must identify a small number of coatings that can provide wear resistance, and the required frictional properties, for components at all temperatures of interest. Those coatings identified to date will meet many of the requirements.

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There is a clear need to produce results under standard conditions and the preliminary tests have provided such information. In further testwork, it will be necessary to expand the standard conditions to include a small number of different loadings for different coatings, to address the conditions of their application. Also, fretting wear must be addressed in reciprocating sliding tests at high frequency.

Consultants to the UK test programme on tribology in the High Temperature Reactor advised measurement of the following characteristics:

1. Friction coefficient at varying loads, speeds and temperatures, both in the initial condition (machined surfaces or other prepared surfaces such as shot-blasted, pickled, etc, which might be applied to sheet metal parts) and after long periods of exposure, at elevated temperature, to the helium atmosphere.

2. Adhesive force after exposure at different temperatures under specific loads for periods of up to at least 1,000 hours.
3. Wear rate under slow reciprocating motion and at different specific loads at different temperatures.

It was deemed possible that initial sorting tests could be readily and economically carried out in high purity helium, but that the final assessment of selected materials should be made in controlled impure helium, simulating as near as practical the reactor atmosphere.

For the UK programme, friction and wear tests in reciprocating sliding contact were carried out at various nominal contact pressures between 0.7 MPa and 6.5 MPa. Typically, a 10 mm pin with a spherical end of 50 mm radius was employed for the tests. These and other test conditions informed the choice of conditions for the initial HTR-E testwork, set out in AMEC NNC's contribution to D40, which can be considered as 'standard conditions' as they have already been applied extensively to sort coated materials for use in helium-cooled HTR systems:

Movement	Reciprocating sliding (pin-on-plate). Stroke $\approx$ 10 mm, frequency $\approx$ 1 Hz, $10^3 - 10^4$ cycles, dwells in some tests.
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Contact pressure	Nominal values (load/pin area) in ranges 0.2 – 1 MPa and 5 – 10 MPa.
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Specimen roughness $R_a \approx 0.4 \mu\text{m}$ or as appropriate.

Temperature	20 – 800°C or as high as achievable.
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Measurements	Dynamic friction coefficient, static friction coefficient during standard tests and after dwells, weight change, wear depth.
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Some consideration should also be given to the pre-exposure of coatings to reactor helium at elevated temperature.

For a compendium of results, it will be necessary to provide information on wear rates in a standard manner. Wear depths, measured by profilometry, will indicate whether a coating has been penetrated, and will allow calculation of wear rates in terms of volume removed per unit load per unit distance of rubbing (m^3/Nm). For the calculation of pin wear rates, in m^3/Nm , weight loss measurements will be required. With wear rates calculated in this way, a direct comparison can be made with results acquired elsewhere.

Appendix 2

DTN/STPA/LTCG/2006-02: Tribology: a compendium of
recommendations – D42

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Titre	Tribology: a compendium of recommendations – D42
Auteur(s)	CACHON L.

Abstract :

Within the framework of the European Program HTR-E, work package 5 on tribology, this document is part of deliverable D42 representing CEA contribution. The aim of this task 3.2 is the redaction of a compendium of recommendations.

Zirconium and CERMET coatings seem to be the best candidates for high temperature helium conditions even if some tribological issues have to be solved, such as the coating adhesion at high temperature.

For temperatures lower than 500°C, there is no real issue to find a couple of material or coating which is suitable for tribology applications in Gas cooled reactor conditions.

Metal like on like tribological contact induces excessive wear by adhesive mechanisms above 500°C. Concerning thin coatings, like TiN or SiC, tribological tests confirmed that they are not adapted to GCR conditions.

Résumé :

Dans le cadre du programme européen HTR-E, WP5 sur la tribologie, ce document est la contribution du CEA sur le livrable D42, dans lequel doit figurer la synthèse du programme HTR-E ainsi que des recommandations en matière de tribologie.

Les revêtements de Zircone et les CERMET semblent être les meilleurs candidats pour les hautes températures, même si quelques problèmes tribologiques restent encore à résoudre, comme par exemple les dilatations différentielles entre le revêtement et le substrat engendrant des problèmes d'adhésion à haute température.

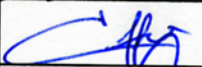
Sous 500°C, les contact homogènes métalliques induisent une usure excessive par des phénomènes d'adhésion.

Concernant les revêtement fins, TiN ou SiC, les essais tribologiques confirment qu'ils ne sont pas adaptés aux conditions GCR.

Mots clés	GCR, tribology, Zirconium, CERMET, SiC
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References

[1] Deliverable 37, HTR-E-02/03-D-5-0-2

[2] Deliverable 41, FRAMATOME contribution, HTR-E- 05/12 – D – 5- 3 – 1 – 2/2

[3] Deliverable 41, CEA contribution, HTR-E- 05/12 – D – 5- 3 – 1 – 1/2

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1. INTRODUCTION.

Within the framework of the European Program HTR-E, work package 5, this document is part of deliverable D42 representing CEA contribution. The aim of this task 3.2 was the redaction of a compendium of recommendations. This document is based on the main conclusions of the D37 [1] and on the results obtained on D41 [2] [3].

2. D37 SYNTHESIS.

The conclusion of D37 [1] can be sent in 3 parts :

- Materials :

Metal surfaces which are in static or sliding contact under the atmospheric and thermal conditions (dry helium up to 1000°C) to be expected in HTR, are subjected to excessive wear or even diffusion welding. Decisive criteria for the load are the temperature, the specific surface pressure, the environmental atmosphere, the surface condition of the contacting parts, and the tendency of the pairings to form alloys. The restraint of movement resulting from the above-mentioned conditions has to be avoided, since under extreme conditions it may result in rupture of the component part and failure of the component. A relatively simple method to protect the surfaces of component parts consists in coating the faces of metal component parts liable to be subjected to wear. The fundamental objective of this method consists in preserving the characteristics of the material (preferably the mechanical characteristics) under noticeable improvement of the surface characteristics.

Coating materials can be classified according to the working temperature ranges :

- For high temperature (higher than 600°C)

The bibliography underlines that a long life time can be expected for ceramic materials such as:

- T up to 700°C $\Rightarrow$ Cr₃C₂/NiCr-Cermets
- T up to 850°C $\Rightarrow$ Cr₂₃C₆-Cr₂₃C₆/NiCr-Duplex
- T up to 950°C $\Rightarrow$ ZrO₂(Y₂O₃)-NiCrAlY

These recommended coatings are similarly suggested by FANP GmbH and CEA.

- For temperatures in the range 180-560°C

From french fast sodium reactor, several coatings were studied. Triballoy 700 (thick coating (100µm–1mm), thin coating with TiC (LSR1 – 20-50µm) and aluminisation (HC17 – 0.15mm) of Stainless Steel gave the best results in this range of temperatures up to 560°C. Massive nickel-base alloy such as PYRAD 53N or CERMET CrC (LC1H) could be used at 560°C.

- Tribology aspect :

Dwell motion and/or fretting have to be studied in the experimental part of WP5, since tribological contact issues are related to these repeated movements. Lubricating coatings have to be enhanced to increase lifetime of surfaces and components (with respect to both dilatation coefficients).

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- Purification of Helium

Bibliography showed that:

- the level of impurities in Helium (O₂, H₂O, H₂, CH₄) and its effect on materials has to be verified since results differ between Dragon, German helium test loops on one side, and Japanese results on the other side.
- water ingress could have serious effect on corrosion and adhesion of the components (control rod failure).

3. D41 SYNTHESIS

The tests program, carried out in the frame of this deliverable [2][3], 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 ₂ O ₃	HR230 + ZrO ₂ /Y ₂ O ₃	500	5	200	1			5MPa = limit load for liner; 900°C for PBMR, same procurement
6			HR230 + ZrO ₂ /Y ₂ O ₃	HR230 + ZrO ₂ /Y ₂ O 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 ₂ O 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 ₃ C ₂	HR230 + Cr ₃ C ₂	500	5	200	1			
18			HR230 + Cr ₃ C ₂	HR230 + Cr ₃ C 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 ₂ O ₃	HR230 + ZrO ₂ /Y ₂ O ₃	500	5	200	1			5MPa = limit load for liner; 900°C for PBMR, same procurement
6			HR230 + ZrO ₂ /Y ₂ O ₃	HR230 + ZrO ₂ /Y ₂ O 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 ₃ C ₂	HR230 + Cr ₃ C ₂	500	5	200	1			
18			HR230 + Cr ₃ C ₂	HR230 + Cr ₃ C 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 », all tests were performed in pure He.

FRAMATOME ANP performed tests at temperatures lower than 500°C. For all tests, the friction coefficient is high (near 0.7), except with the add of solid lubricant in the Zirconia coating where the coefficient of friction decreased to 0,4. The pin on disk tribometer doesn't represent a reel contact (it's a "standard" contact), but it allows to characterize couple of materials easily and rapidly. It is useful in the study of coatings for example, to evaluate the adhesion of the deposit... In some

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cases, the wear is important, particularly in contacts metal/metal. With zirconia coatings, the surfaces are few worn. The tungsten carbide coating is resistant to wear, but the counterpart is well worn. The TiC coating was too thin to be correctly characterized. It could be interesting to test another deposition process (like PACVD).

CEA performed tests at high temperatures, from 500°C to 900°C, which allowed to find the limit of the operating conditions of materials. The CEA tribometer is a “pins on rail alternative sliding” tribometer, simulating a plane / plane contact. The main conclusions for each test are (table 2) :

- **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** ($\text{Cr}_3\text{C}_2 - \text{NiCr}$) : The tests were performed at 700°C and 5 MPa of contact load. This is the single test, in this program, for which the 5000 required cycles were performed. The tribological behavior is very good, with a wear rate of 0.008 $\mu\text{m}/\text{cycle}$ and a coefficient of friction lower than 0.3.
- **Zirconium coatings** :
 - **ZrO₂-Y₂O₃ / NiCrAlY** (Classic plasma spraying) : the test was performed at 800°C and 5 MPa of contact load. Only 1600 cycles were performed because of the coating destruction. The wear and the coefficient of friction ($\mu=0.6$) are too high.
 - **75% (ZrO₂-Y₂O₃) + 25% CaF₂ / NiCrAlY** (Classic plasma spraying) : the test was performed at 900°C and 5 MPa of contact load. The wear and the coefficient of friction ($\mu=0.3$) were low, but the thermal expansion between the coating and the substrate induced delamination of the coating.

To improve the tribological behavior of the zirconium coating, the use of solid lubricant like CaF₂ is very efficient, even if the differential thermal expansion between the substrate and the coating induces its delamination. The improvement of the bond between the coating and the substrate and of 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 load. The coating is too thin and too brittle, and the substrate too soft at 900°C to withstand the load of 5 MPa. This induces the failure and the destruction of the coating.

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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 ₂ O ₃ (PLASMA SPRAYING)	800	5	0,65±0,1	1500	-	
[ZrO ₂ -Y ₂ O 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 ₃ C 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 2 : Synthesis of CEA tribological results obtained in the frame of HTRE – WP5 European program.

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4. COMPENDIUM OF RECOMMENDATIONS

According to the results found in literature, reactors feed back and the last tests carried out in the frame of the European Program HTR-E, Zirconium and CERMET coatings seem to be the best candidates for high temperature helium conditions :

- For Zirconium coatings, the add of solid lubricant improves the global tribological behavior (less wear and coefficient of friction about 0,25)
- For CERMET coatings, especially chromium carbide grade, their tribological behavior is also very good ($\mu < 0,3$, and low wear rate). Tests performed at lower temperature, on WC CERMET, shown its good wear resistance but a high coefficient of friction.

Nevertheless, tribological tests shown different issues :

- For Zirconium coatings, with standard low pressure plasma processes, the density of the coating has a major influence, but in opposed way, on its wear resistance and on its bond to the substrate : a mean density allows a correct bond to the substrate but a bad wear resistance at low contact load at high temperature, and inversely, a high density allows a high wear resistance but the coating becomes stiff and brittle and the bond with the substrate was embrittled. So a trade off 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 processes (like HVOF), and / or nanostructured powder could increase the coating density with increasing the bond to the substrate. This way has to be studied in order to improve both the wear resistance and the bond with the substrate of zirconium coatings.

- For CERMET coatings, 700°C-800°C seems to be a temperature limit to have a correct coating adhesion on the substrate. Tests have to be performed to find this limit temperature. On the other hand, the use of a thermal accommodation underlayer like NiCrAlY could be suitable to avoid coating delamination at high temperature. Another interesting way to be studied is the efficiency of the add of solid lubricant in CERMET coatings in order to limit the coefficient of friction which can be high, i.e. $\mu = 0,7$ for WC CERMET.

For lower temperatures (<500°C), if there is no requirement on the coefficient of friction, all contacts, with at least one thick coating on a counterface (thickness > 200µm), tested by FRAMATOME gave a good behavior in wear resistance. If there is a need to improve the friction behavior of contact, solid lubricant coatings have to be used and evaluated in experimental programs.

Metal like on like tribological contact induce adhesive wear then ploughing / adhesion and at the end an excessive wear.

Concerning thin coatings, like TiN or SiC, tribological tests confirmed that they are not adapted to HTR conditions.