EUROPEAN COMMISSION
5th EURATOM FRAMEWORK PROGRAMME 1998-2002
KEY ACTION : NUCLEAR FISSION

HTR-E

High-Temperature Reactor Components and Systems

CONTRACT N°
FIKI-CT-2001-00177

Deliverable D39 for " Tribology in Helium Environment"

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FRAMATOME -ANP

FRANCE

Dissemination level : RE
Document N°: HTR-E- 03/12 – D – 5- 1 - 3

Status : Preliminary
Deliverable D39
Framatome ANP identification N° :NFTW – R/03.898A

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N° NFTW R03.898 A

MODIFICATION	
INDICE	DATE
0	30/01/2004

HTR-E / WP5

DELIVERABLE D39
REVIEW OF EXISTING MATERIALS AND COATINGS

MOTS CLES

Tribology
High temperature
Gas environment– helium
Material
Coatings
AGR – Dragon - aerospace

DISTRIBUTION

Niveau de protection (1)

~~**NORM** (normal)~~

FILT (filtré)

~~**CONF** (confidentiel)~~

N° UD : 72H.AFM0088

Nature document : NT (2)

DIFFUSION	FONCTION	NOM	VISA	DATE
E. BREUIL (NFPVEI) D. BUCKTHORPE (NNC) D. HITTNER – C. RICHEZ (NZ) C. FALCAND (FANP-NFTM) D.KACZOROWSKI (NFTC)	Rédacteur	I. BOBIN (NFTW)	[signature]	2004/01/15
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Modifications

Revision	Date	Modifications
0	30th January 2004	First issue

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(1) European contract: "High temperature reactor components and systems" – Project FIKI – CT-2001-20177 "HTR-E" – August 2001 – Annex 1

(2) WP5 Work Plan Report for : " Tribology in Helium Environment" - Document N°: HTR-E- 02/03 – D – 5- 0 – 1 – deliverable D36

(3) Deliverable D38 – HTR-E 02/12-D-5-1-2 –rev B - Review and identification of typical HTR tyribology conditions of components – 11th/08/2003

(4) Contribution from NNC to Deliverable D39 – C6940/TR/0004 REV1 – May 2003 – B. RILEY (NNC)

(5) Deliverable D37 – HTR-E- 02/12 – D – 5 – 1 - 1– Review and identification of tribological problems in former HTR or FBRs

(6) Summary of contribution from FZJ to Deliverable D39 – PJ ENNIS – 10th October 2003

(7) Contribution from FANP to Deliverable D39 – TFCM R03.1059 – C. FALCAND -

SUMMARY - CONCLUSIONS

A number of materials including Stainless Steel 316, other austenitic steels type 347, alloy 800, carbon steel, 9Cr steel, or nickel alloys and graphite, are envisaged in PBMR and GTMHR for several main components such as control rod drive mechanisms, hot gas duct, stators seals, internals. To ensure reliable operation surface coatings will be needed to provide additional protection against damage caused by wear, fretting, etc. on certain reactor and PCU components. Possible coatings have been short listed from past experience on HTR's (Dragon, HTR Module) and from component experience on the AGR.

The temperature ranges considered are:

- 100-500°C
- 500-800°C
- above 800°C

Solutions were found by using coatings just as carbide based cermets, or lubricants such as MoS_2 cermet to limit wear and rubbing in CO_2 (for AGR) or Helium atmosphere (Dragon, German module). The same or other types of coatings were also found in the turbine or aerospace industry operating in oxidising atmospheres. Several examples were also found of metallic couples causing grooves or scratches and material transfer due to bad tribological properties.

The results for the best possible coatings as a function of environment and temperature range have been summarised into two tables (Tables B & C) for sliding and fretting actions.

From the above investigations a list of reference coatings for use in helium as a function of temperature range has been established.

HTR-E / WP5: DELIVERABLE D39
REVIEW OF EXISTING MATERIALS AND COATINGS

INTRODUCTION

This report covers a review and identification of materials and coatings used in former HTRs such as the Dragon reactor (UK), and Advanced Gas–Cooled Reactor (AGR) in UK, tested for the German HTR Nuclear Process heat (PNP) project in Germany, or used in other industries such as aerospace field. The report corresponds to the deliverable D39 of the HTR-E Project [1, 2].

The rubbing behaviour of some materials without any coating is given in the report from PNP experience, but the tribological results which are given concerned essentially coatings.
Coatings are used to confer suitable tribological properties to materials such as principally, austenitic stainless steel and mild steel for AGR, space or tool industry, Nickel superalloys in PNP project, or space projects. Many coatings can be incorporated at the design stage and others are introduced to overcome specific problems.

The purpose of this document is to provide wherever possible available information on the materials and coatings used and the different environmental and tribological conditions (type of contact, contact conditions, loading conditions) experienced. The results from this work will assist in establishing a first classification of materials and coatings before further tests are undertaken during next step of HTR-E (task 3).

This document contains the results of work of NNC and FANP on Deliverable D39 of the HTR-E Project. The summary provided by FZJ is given as an annex. FZJ complete contribution will be attached later.

This document is built as follows:

- presentation of possible problems in PBMR and GTMHR
- materials used for close components in AGR, Dragon, PNP project, other industries for different temperature conditions
- tests of coatings in these temperature conditions, and results
- recommendation of coatings for 3 ranges of temperatures: under 500°C, in the range 500-800°C, temperatures upper than 800°C.

1. Possible material problems in PBMR and GTMHR (D38 ref [3])

These problems were reviewed in the D38 deliverable. They are recalled here under:

1.1. PBMR

A table of potential problems is given in annex (table 1). The envisaged materials for PBMR are Stainless Steel 316, other austenitic steels type 347, alloy 800 or similar, carbon steel, … but several tribological problems are also foreseen: static adhesion on valves at 150°C, fretting on heat exchangers (adhesive wear) or bearings, sliding with contact adhesion on drive mechanism in the range 450-900°C, ….

1.2. GTMHR

For GTMHR, the review has shown the following materials and type of motion to be sustained by the components:

- Control rod drive mechanisms: sliding and bearing on 38CD4 surfaces under 595 to 1030MPa in the range 45-490°C
- Hot gas duct: sliding or fretting on CrNi55MoWZr alloy at 500°C
- Stator seals: fretting on ChS57-VI or EP99 materials at 100-500°C
- Internals: fretting on Nickel alloy or 9Cr steel, and graphite at 450-800°C

1.3. In summary

The envisaged materials are Stainless Steel 316, other austenitic steels type 347, alloy 800, carbon steel, 9Cr steel, or nickel alloys and graphite.
No coating is proposed, although they are mainly austenitic steels, that suffer from poor tribological performance.

The temperature ranges are:

- 100-500°C
- 500-800°C
- upper than 800°C

2. Review of materials and coatings used in AGR (NNC [4])

The AGR is a development of the Magnox system in UK, designed to raise the maximum temperature of the gas coolant by some 300°C, and thus improve the thermal efficiency and the economic performance of the power station.

CO_2 gas was selected as the coolant because of its low cost and availability, good heat transfer performance including natural convection, good depressurisation characteristics, good neutronic properties, its compatibility with graphite and fuel cladding. Coolant chemistry control was important for corrosion control in AGRs and detailed knowledge of the coolant composition and impurity level targets vital for determining the friction and wear characteristics of all the metallic materials in contact.

2.1. Review of materials

A list of materials used in the AGR reactor for components working at temperatures upper than 300°C is given in **table 2**. A more detailed description of metallic materials used for various parts of components, is given in NNC report as tables 1a to 1f, and table 2 for tubes.

In these NNC tables and in table 2 of the present report as a summary, it appears that the major part of the listed components is made of Cr-Ni Austenitic Steel such as alloys 316, 321 or 347, and were working between 430 and 650°C. It concerned control rod joints, boiler unit,

superheater unit, reheater unit, guide tubes and gag unit. Nickel alloy such as Nimonic was used for superheater at 590°C. Mild steel, 1Cr or 9Cr steels were also used for boiler tubes.
In some cases (control rod joints, boiler unit, guide tubes, gag unit), a coating was needed. The used coatings were coatings such as:

- **Cermet type (Cr_2C_3 base** bound together by a small amount of a softer, metallic phase such as NiCr binder, and sometimes mixed with WC_3) – called LC-1B if deposited by D-Gun, LC-2 when plasma gun deposited, and LW-5 when mixed with WC_3
- **Metallic nickel based coatings** called Alloy C, alloy 50 or alloy 60 according to the composition of the alloy. These alloys were TIG welded, cast or spray fused.

They are detailed in **Table 3**.

2.2. Review of deposition techniques

2.2.1. Detonation-Gun, or D-Gun coatings

They were used to deposit **LC-1B** and **LW-5** cermet coatings. They were applied using a device similar to a rifle barrel, into which powdered coating material and an oxygen/acetylene gas mixture are introduced and then ignited by a spark discharge. The mixture detonates and the powder is propelled from the barrel at high velocity. The operating cycle is repeated many times to build up the coating.

Advantages of the technique are:

- High density deposits
- Good adhesion to the substrate
- Minimal surface preparation
- Low heat input to the substrate material.

Disadvantages are:

- Unsuitable for some geometries, e.g. inside tubes
- One supplier : Union Carbide (now Praxair)

2.2.2. Plasma Spray coatings

In this technique, powder is carried to the component surface in a high temperature stream of inert gas.

Advantages:

- A wide range of coating types may be applied
- Deposition on tube bores is standard

Disadvantages:

- Porosity greater and adhesion to substrate poorer than for D-Gun coatings

The only coating applied using this technique was **LC-2** (from Union Carbide), which is similar in composition to LC-1B.

2.2.3. Spray-Fuse coatings

In this flame spraying technique, powder is deposited from an oxy-acetylene flame and further heating is supplied to fuse the deposit.

Advantages:
- Good adhesion to substrate
- Thick coatings (several mm) possible
- Good apparatus mobility

Disadvantages:
- High degree of substrate heating
- Post-deposition machining required
- Poor quality control

Coatings **SF50** and **SF60**, from Deloro Stellite, were applied by the spray-fuse method. Both coatings are nickel-based and also contain chromium, iron, silicon, boron and carbon.

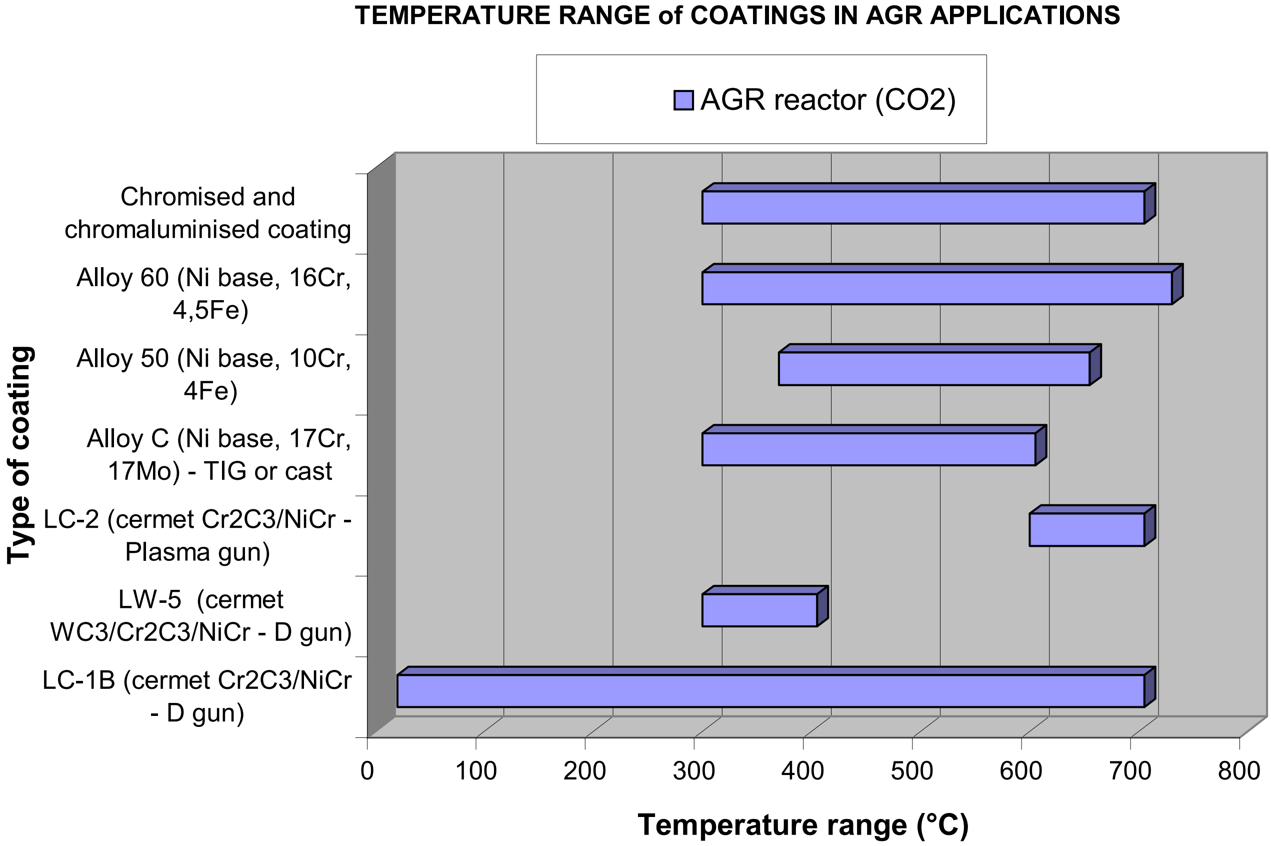
2.2.4. Weld deposits

The technique normally used was that of transferred arc inert gas (TIG) welding. The advantages and disadvantages of this technique are similar to those of spray-fused coatings. Nickel-based Alloy C, Alloy 50 and Alloy 60, from Deloro Stellite, were used in AGRs. Alloy C is of high chromium and molybdenum content, with some iron and tungsten. Alloy 50 and Alloy 60 are similar in composition to SF50 and SF60.

2.3. *Review of the deposits applied in AGR*

Table 4 gives AGR coatings and operating temperatures between 300 and 750°C. The base material is indicated together with the corresponding component.

Tables 5A and 5B complete the information by giving respectively for temperatures up to 400°C and up to 725°C, the coated components with their counterface when possible, and the type of actions which have to be sustained by the surface of the components. It is generally sliding and fretting, with some localised case of bearing.
Generally speaking, the coatings were applied in the following ranges of temperatures. Their results in rubbing are given in the next paragraph.



3. Tribological results of coatings used in AGR (NNC [4])

3.1. Tribological behaviour of these coatings

The tribological behaviour of the coatings listed before was examined in regards with several criteria:

3.1.1. Spalling:

Spalling may result from a combination of factors, including internal stress resulting from the coating process, thermal stress due to temperature cycling and differential thermal expansion, oxidation, poor adhesion of the coating to the substrate and weakened adhesion as a result of coating/substrate interface corrosion. The nickel-based hard coatings were deemed to be better than the cermets with regard to these compatibility issues. Nevertheless, corrosion testing of LC-1B over many thousands of hours did not reveal a serious spalling problem.

- **Ni based hard coatings considered better than Cermets**
- **Corrosion testing on LC-1B did not reveal any problems over many thousands of hours**

3.1.2. Oxidation

- **LC-1B & LC-2 – Tests at 600°C were satisfactory**
- **At 700°C, LC-2 showed some detachment on corners after 10 000h. No problem elsewhere. No problem on LC-1.**
- **At 700°C, LC-1B & LC-2 showed oxidation of coating but not of the substrate.**

- LW-5 oxidation of Tungsten Carbide - tested at 300 & 400°C (some deterioration): To be used at temperature up to 350°C on mild steel and up to 400°C on Stainless Steel.
- **SF50, SF60 (both poor) & Alloy C (good at 550°C, blistering at 600°C)**

- Chromised coatings
  - on mild steel - tests at 400°C – blistering after 86 000 hours
  - on 316 Stainless steel - good after 10 000h at 700°C.

3.1.3. Nuclear compatibility

It concerns mainly Boron, which is present in Alloys 50 & 60, but finally these coatings were not used in the AGR gas circuit

3.1.4. Friction, static adhesion and wear

Where coated components are in continuous or intermittent contact with other components and there is relative movement between the parts, seizure or malfunction will be governed by the value of friction coefficient. However, no single value of friction coefficient can be attributed to a material pair for this parameter because it will change with temperature, loading, sliding speed, frequency of vibrational relative motion, etc. Also, two aspects must be considered: dynamic friction and static adhesion.

A great amount of friction testing, at various temperatures and gas pressures, and under different loadings, was carried out in support of the AGR programme.

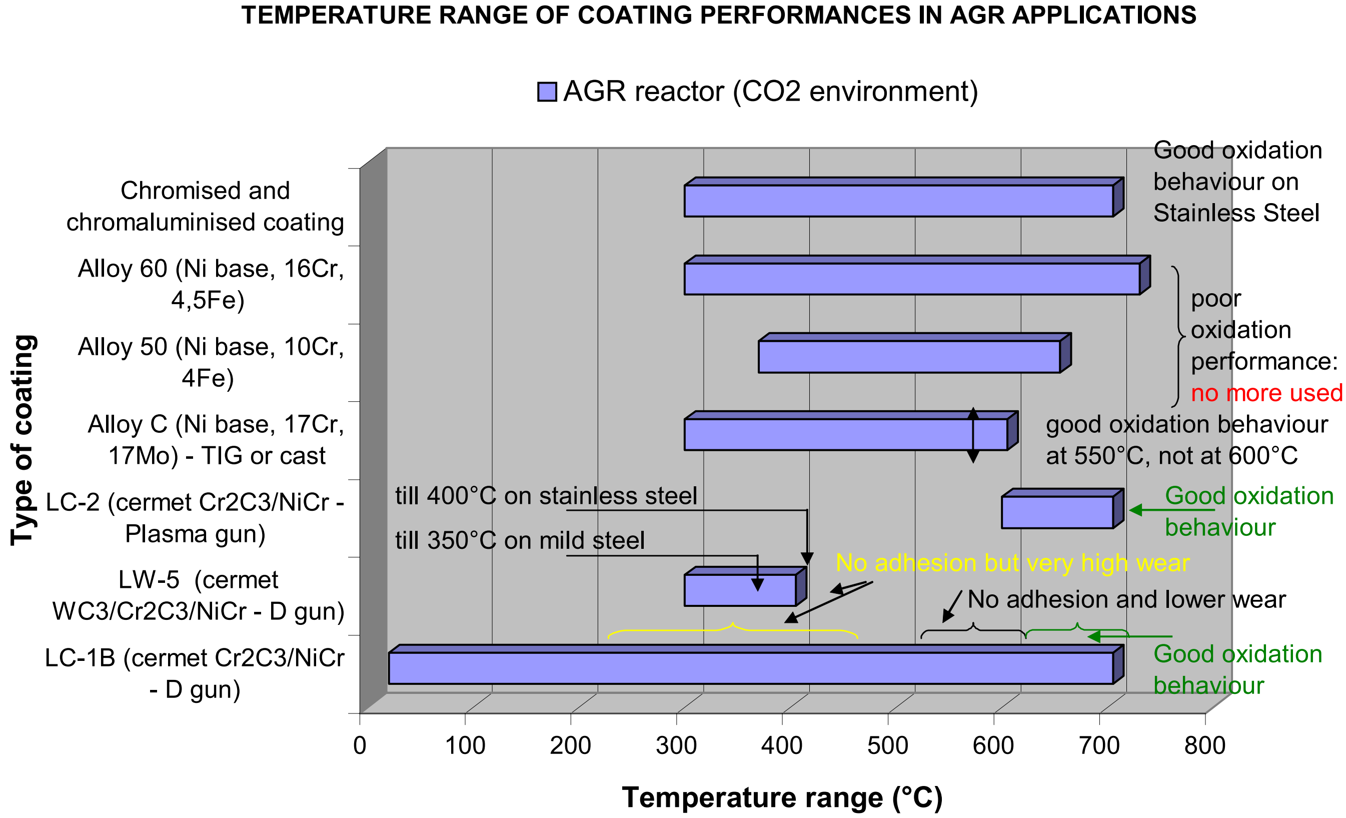
Mild and low carbon steels are susceptible to adhesion in carbon dioxide at temperatures of 300°C and above, while austenitic stainless steels will resist adhesion at temperatures up to approximately 550°C. Adhesion occurs largely as a result of the growing together of surface oxides but also by welding at metallic junctions. Hard coatings may be applied to overcome the risk of adhesion by protecting the underlying metal from oxidation.

- Adhesion trials were done at temperatures up to 650°C in CO_2 environment
- Best were LC-1B, LW-5, Spray Fused SF60 for mild steel and Stainless Steel against adhesion
- Wear rates for LC-1B & LW-5 worst in the range 200-450°C; and lowest at 500-600°C

- Wear rates in CO_2 dependent on temperature, lower rates occur in high pressure gas

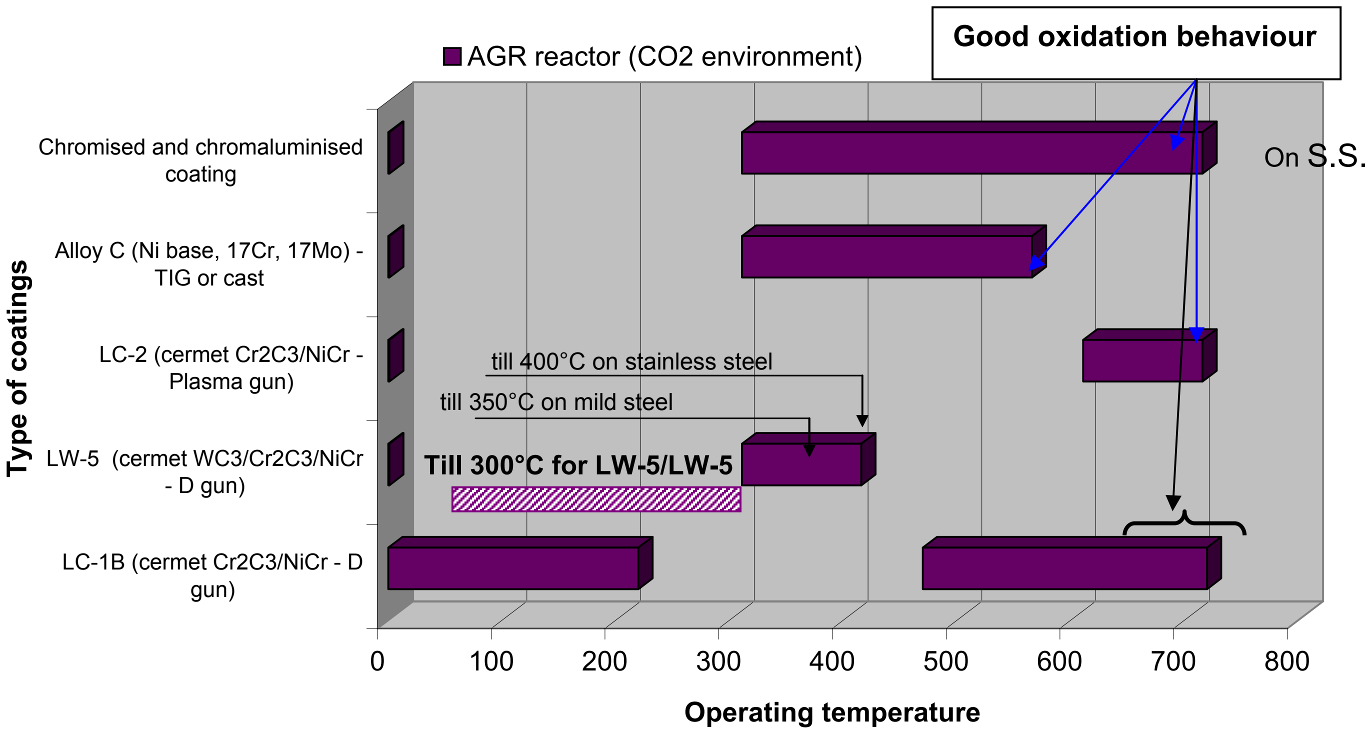
3.2. Tribological behaviour : summary

The initial graphic of paragraph 3.3 can be annotated as follows, for CO_2 environment:



It results in the following simplified one:

TEMPERATURE RANGE OF COATING PERFORMANCES IN AGR APPLICATIONS (NNC)



With:

LC-1B and LC-2 : Low substrate heating during deposition,
: Surface finishing usually nor required
: Good resistance to oxidation and to static adhesion

LW-5 : Low substrate heating during deposition,
: Surface finishing usually nor required

Alloy C : High substrate heating during deposition,
: Post deposition finishing required,
: Fairly good resistance to oxidation,
: High wear rates and rough galled surfaces

4. Review of materials and coatings used in Dragon (NNC [4])

4.1. Introduction of Dragon experience (NNC) [D37 [5]]

The OECD High Temperature Reactor Project (Dragon) ran from 1959 to 1976. Because potential tribological problems resulting from operation in relatively pure helium were recognised early, design solutions were, in general, found. Since wear and adhesion become more of a problem as the temperature increases, temperatures were kept low wherever possible.

Finally, few tribological problems arose during the operation of the Dragon reactor. Wherever possible, potential problems were designed out by placing rubbing surfaces and mechanisms in low temperature areas. To address issues arising from, for example, charge/discharge operations, thermal cycling and vibration, a programme of testing was carried out. Suitable materials combinations were identified for many different applications (see **Table 6**).

It was noted :

- The effect of dwelling (dwell sliding or fretting)
- The effect of the temperature
- The low effect of impurities in helium environment
- No effect of helium pressure

4.2. Tests in the frame of Dragon (NNC) [D39-[4]]

4.2.1. Friction and wear

2 programs were realised:

Friction and wear – PROGRAM N°1 (table 7)

Tests type 1: in pure helium at 0.1 MPa or 5 MPa pressure,

Tests type 2: in impure (reactor) helium at 2 MPa, at temperatures from 20°C to 800°C. The partial pressures of the impurities in the reactor helium were as follows. H_2O: 5 Pa, H_2: 50 Pa and CO: 50 Pa.

Contact pressures of 2.1 MPa and 6.5 MPa were employed.

The following conclusions were drawn from the reciprocating sliding tests:

- In pure helium, no systematic effect of gas pressure was observed. No significant or systematic difference was observed between wear behaviour in pure helium and in reactor gas, **but concentration of water and O_2 had considerable effect on the friction coefficient.**
- All the parameters varied during the tests, temperature had by far the greatest effect, though this was not necessarily the same on both coefficient of friction and wear for the different material combinations.
- There was no systematic effect of contact pressure on friction but when wear was high a significant increase with contact pressure was apparent.
- No materials combination behaved well under all test conditions.

- No combination behaved entirely satisfactorily at 20°C.

Friction and wear – PROGRAM N°2 (table 8)

Friction & Wear: - Dwell Tests - in-reactor conditions , 450-750°C, 100 cycles /day & 10 cycles/day – 20 days

Results from a further programme of work on alloy couples endorsed the above conclusions.

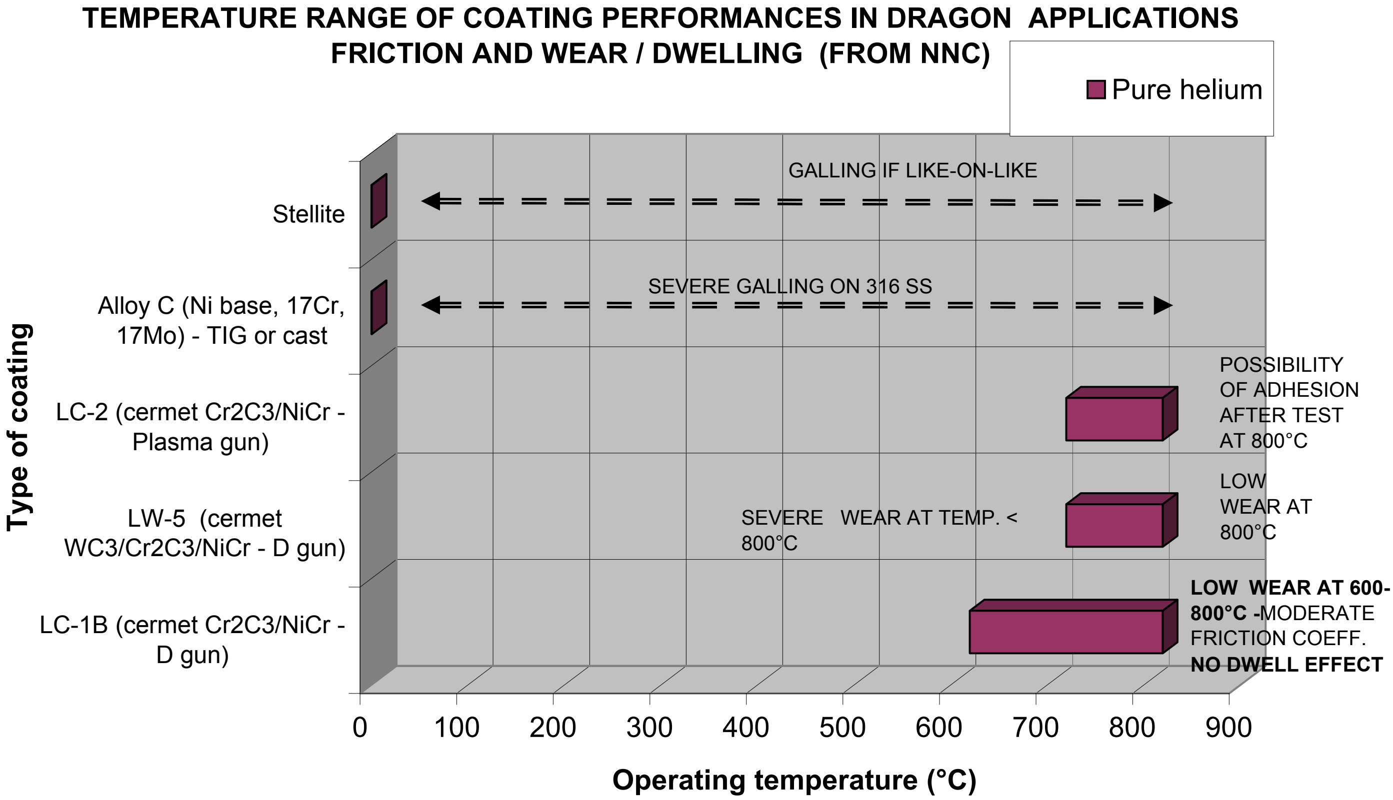
They also showed that the concentration of water, and more significantly, oxygen in the helium had a considerable influence on the coefficient of friction. This was attributed to the formation of lubricating oxide films.

In a HTR, some rubbing interfaces associated with reactor components and structures will move only during temperature cycles or when components are inserted or withdrawn from the reactor. To assess the effect of a dwell, a serie of tests was performed in reactor-composition helium at temperatures between 450°C and 750°C on seven materials combinations. Each combination was tested in low speed reciprocating relative motion for 100 cycles and the results were compared with those from tests comprising 5 cycles each day for 20 days. Tests were performed on the following combinations:

Alloy C	AISI 316
AISI 316	AISI 316
Nimonic 90	AISI 316
Nimonic 90	Nimonic 90
Chromium carbide LC-1B	Chromium carbide LC-1B
AISI 316	EYC9106 Graphite
En40B	En40B

General conclusion:

- Friction coefficient similar with & without dwell;
- Wear, generally in material transfer form, caused by welding during dwells ;
- Chromium carbide LC-1B, like-on-like, and AISI 316 v EYC9106 (graphite) also showed no effect of dwell on load. (see table 8) ;
- The remainder of the materials pairs, though, showed more severe wear in the dwell tests than in the continuous tests (tests at 650-750°C).



4.2.2. Fretting

Impact sliding; 350 and 750°C, 75Hz and 150Hz, dry He & wet gas.

- Wear rates similar and within 10^{-13} to 10^{-12} $m^3.kg^{-1}. m^{-1}$
- **Wear rates marginally greater in pure gas at 750°C, than in impure gas**
- Wear rates marginally greater in impure gas at 350°C, compared to 750°C
- Welding after 100h duration test.

4.2.3. Static adhesion

The static adhesion of various potential HTR materials was assessed at temperatures in the range 400-800°C in the 'pure' and 'impure' helium atmospheres used for the fretting tests. Materials included stainless steels, mild and low alloy steels, Nimonic alloys, Incoloy 800, hard facing alloys and graphites, alumina and flame sprayed alloys. Dead weight loading was applied to produce an interfacial pressure of 0.3 MPa. Specimen pairs were parted in tension after cooling to ambient temperature in helium.

The results indicated:

- Little or no adhesion of materials at 400°C in either of the two atmospheres
- Between 650°C and 800°C all metallic pairs examined showed adhesion with the exception of Nimonic 90 v En 58J stainless steel.
- At 650°C adhesion was comparable with that found in carbon dioxide.
- With increasing temperature, a much higher degree of adhesion was found. The most powerful static adhesion was found in self couples of En 58J stainless steel.
- After 1000 h exposure at 800°C a parting pressure of 7 MPa was required on En 58J stainless steel.

5. Review of coatings applied to gas turbine components (NNC- D39 [4])

Hard coatings, mostly applied by Detonation-Gun and plasma spraying, are used on hundreds of parts in production aircraft gas turbines.
The different cases listed by NNC for aerospace applications are given in tables 9 and 10 for temperatures respectively less and upper than 540°C.

In aerospace applications, the environment is oxidising gas. In this environment, the tribological solicitations are described as:

Up to 540°C:

- ***Sliding***, using Chromium carbide /NiCr (like coatings LC-1B and LC-2 in AGR and Dragon) – *W-carbide with Cobalt is widely used but not nuclear compatible*
- ***Fretting***, using W-carbide with Cobalt
- ***Against galling***, using Copper / Nickel / Indium alloy
- ***Against particle erosion***, using W-Titanium carbide + Nickel, Titanium nitride (*or W-carbide with Cobalt*)
- ***Against rubbing***, using aluminium oxide and Chromium carbide /NiCr (like coatings LC-1B and LC-2), (*or W-carbide with Cobalt*)

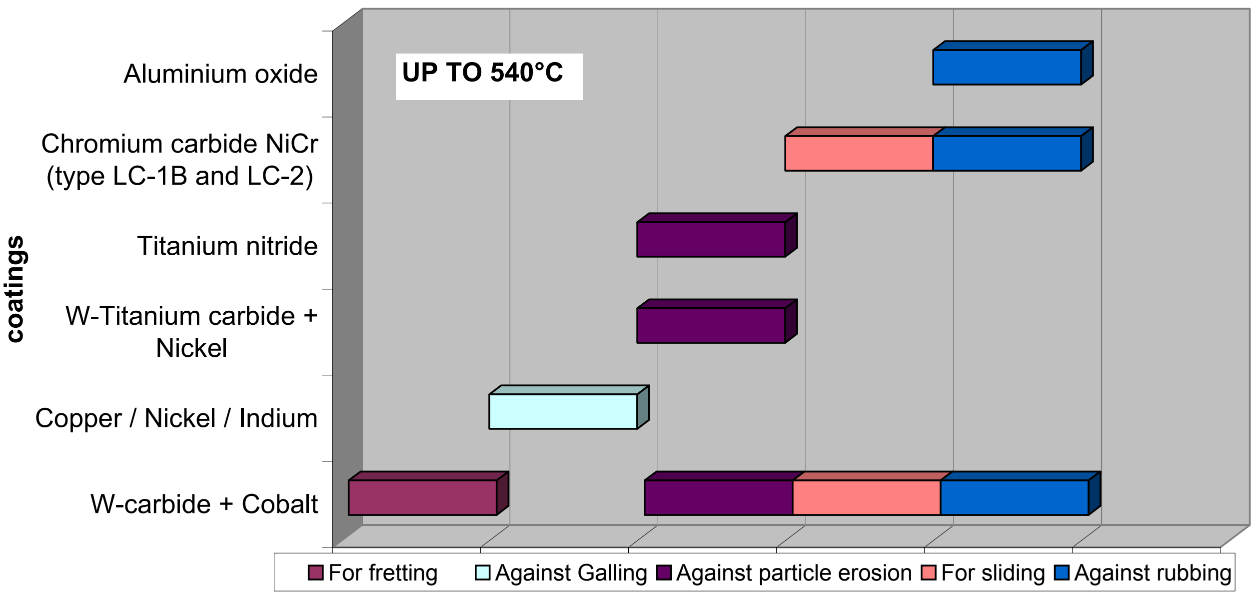
For temperatures upper than 550°C:

- ***Fretting***, using Chromium carbide /NiCr (like coatings LC-1B and LC-2), Cobalt alloy cermet with Al_2O_3 dispersion, (*or W-carbide with Cobalt*)
- ***Against loosening***, using Copper / Nickel / Indium alloy

- ***For thermal barrier with erosion effect or not***, using Duplex MCrAlY stabilised Zirconia
- ***Against rubbing***, using Cobalt alloy cermet with Al_2O_3 dispersion (*or Cobalt alloy*)

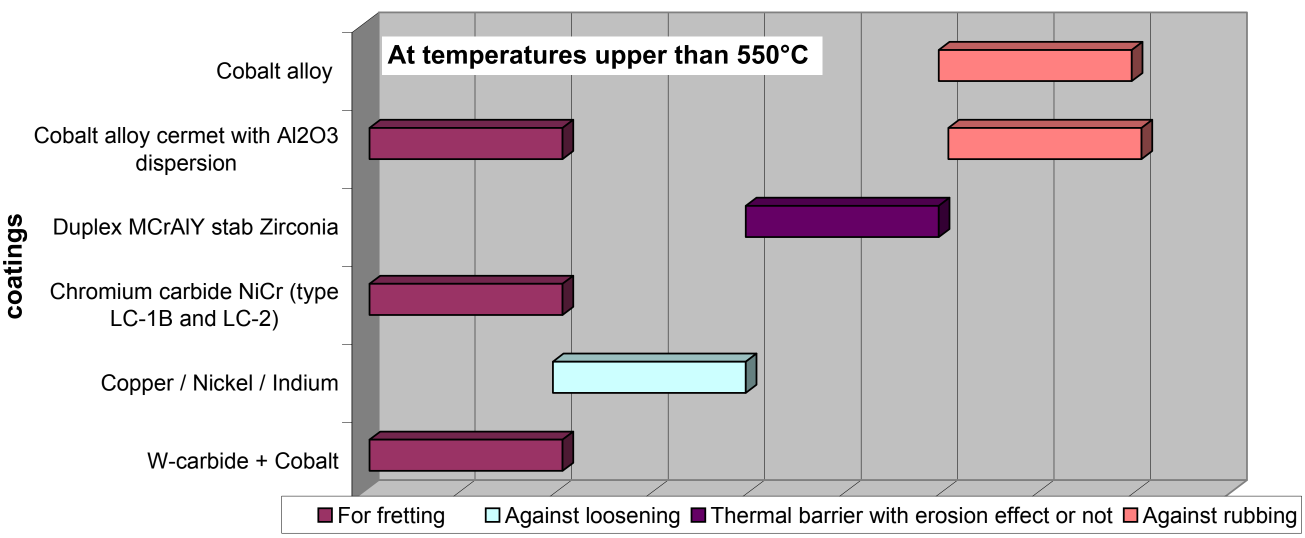
The aptitudes of each coating are visualised with the following diagrams:

Coatings applied to turbine components at temperatures up to 540°C



Tribological solicitations

Coatings applied on gas turbine components at temperatures upper than 550°C



Tribological solicitations

6. Review of materials and coatings used in PNP project (FZJ summary – [6])

The main surface treatments and coatings which are presented concern the work on anti-friction and wear resistance of various critical PNP plant components that undergo sliding contact, such as thermal expansion joints and valves or are subject to fretting as a result of vibrations.
This summary is restricted to the friction and wear behaviour which were of significance for the modular HTR components (summary available in annex).
The friction and wear behaviour of PNP components may be divided into three temperature regions: below 700°C, in the range 700-850°C, and above 850°C.

6.1. below 700°C

In this range of temperatures, the metallic combinations with no coating which were working without any galling were the following:

SLIDING

- Inconel 625 / Inconel 625 at **700°C** in Helium atmosphere (HHT-load 20MPa)
- Colmonoy 6 / Colmonoy 6 at **600°C** in Helium atmosphere (HHT- load 50MPa)
- Triballoy 700 / Triballoy at **600°C** in Helium atmosphere (HHT- load 50MPa)
- Boiler plate material H11/H11 at **400°C** in Helium atmosphere (HHT-0.05MPa)
- INC713LC/X20CrMoV 12-1 at **350°C** in Helium atmosphere (HHT-load 10MPa)

FRETTING

- 13CrMo4-4 / 13CrMo4-4 at 500°C (less wear for boiler tube material)

Conclusion at T<700°C:

For major of unprotected metals which were tested (Nimonic 75, 17CrMo 5-5, and like on like: Inconel 617, stellite 6, St37, Hastelloy X, Nb alloy, … or other combinations of previous alloys (Inc 625, …), ***significant wear occurred in Helium atmosphere at low temperatures: need of coating***

- TiC coating : low friction coefficients in pure Helium (T<600°C).

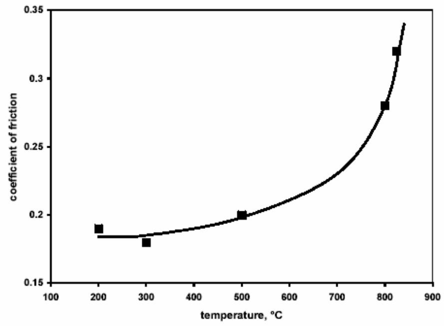


Figure 3
Temperature dependence of the coefficient of friction of TiC in helium (4.6)

6.2. Friction and wear properties of PNP alloys at 700 - 850°C

In this range of temperatures, several metallic combinations with no coating were tested.
In pure helium, the only combination which was not leading to galling was:

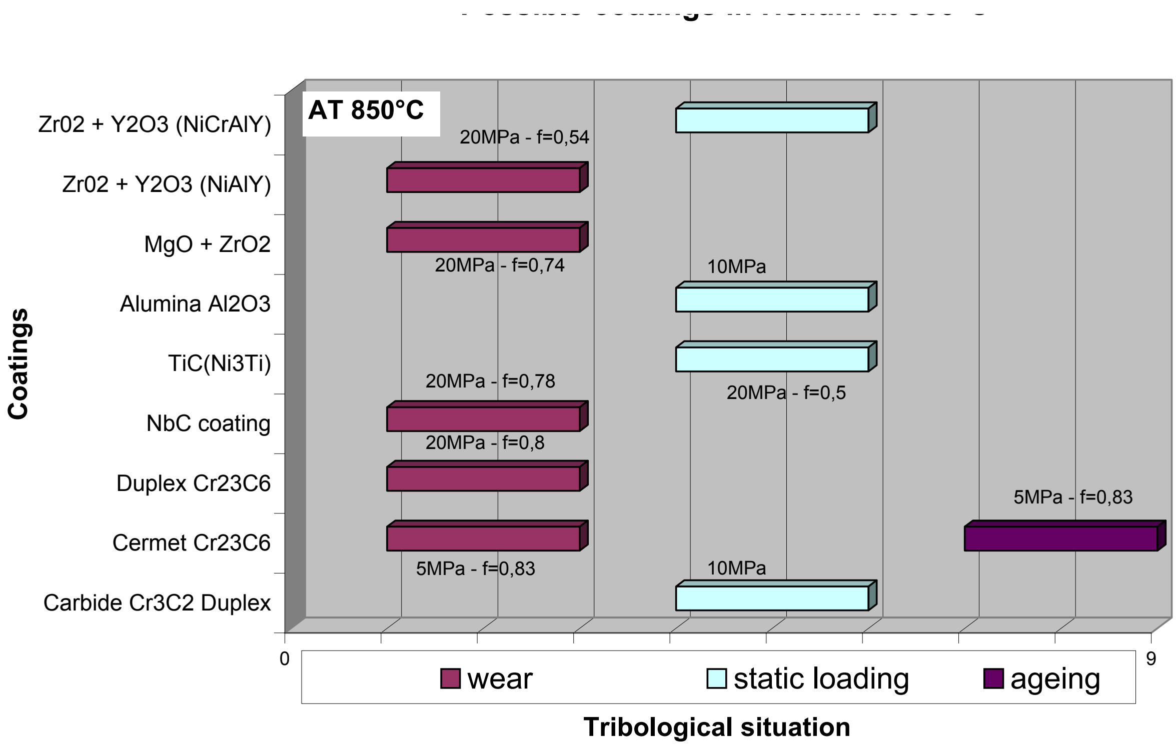
- Hastelloy X / Inconel 601 which did not show any galling after 100 hours at 800°C, and slight galling after 500 hours (japan study)

Any other combination with Hastelloy X, Incoloy 800, Inconel 601 and 625 wore in pure Helium.
It was shown that without any surface protection, wear with plastic deformation of the mating surfaces was detected at 800°C. For Hastelloy X/Hastelloy X, it was determined that a protective coating was required above 700°C, but no coating was suggested, except a carbide coating tested by General Atomic, under HTR conditions at 816°C: $Cr_{23}C_6$ or Cr_3C_2 coating (type of binder?).
A ZrO2 coating on Hastelloy X strongly lowered the friction coefficient in Helium, from 800 to 1000°C.
It was noted elsewhere that at 700°C:

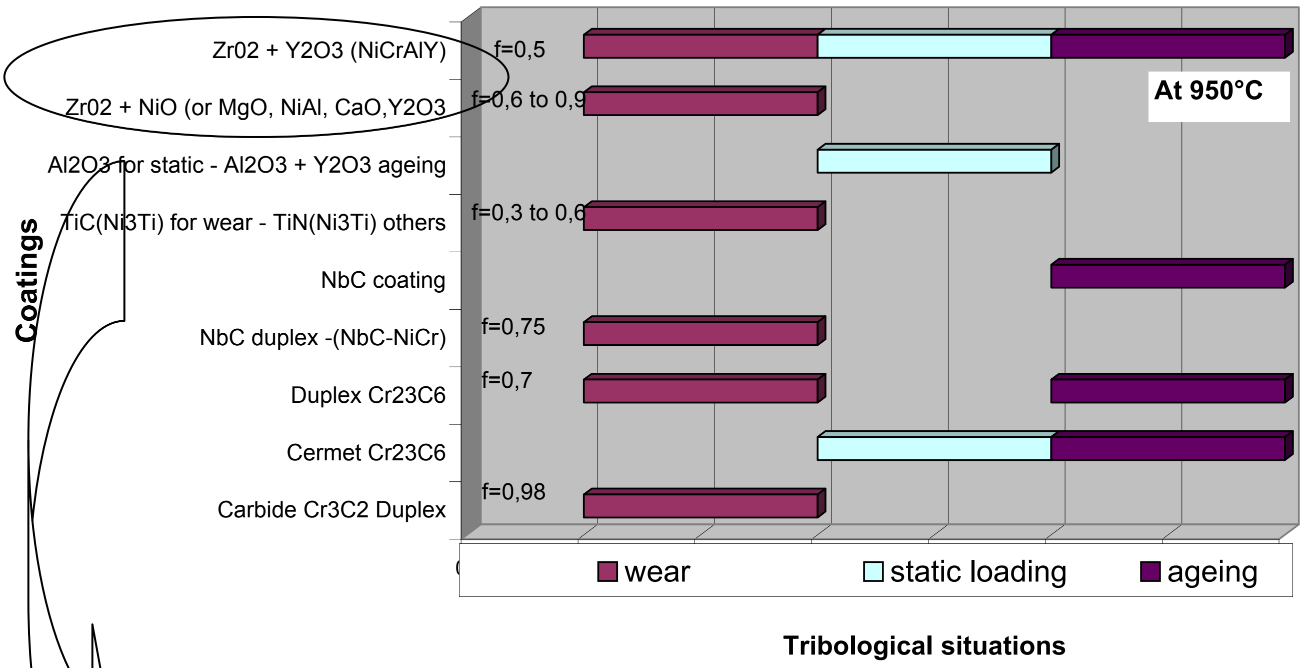
- Cr_3C_2 / NiCr gave good results, and cermet $Cr_{23}C_6$ bad results (D gun deposited).
- Carbide coatings are not adherent on Alloy 800

6.3. Friction and wear properties of PNP alloys above 850°C

A selection was made from all the available tables. The following diagrams give only the coatings with positive results, per temperature range.

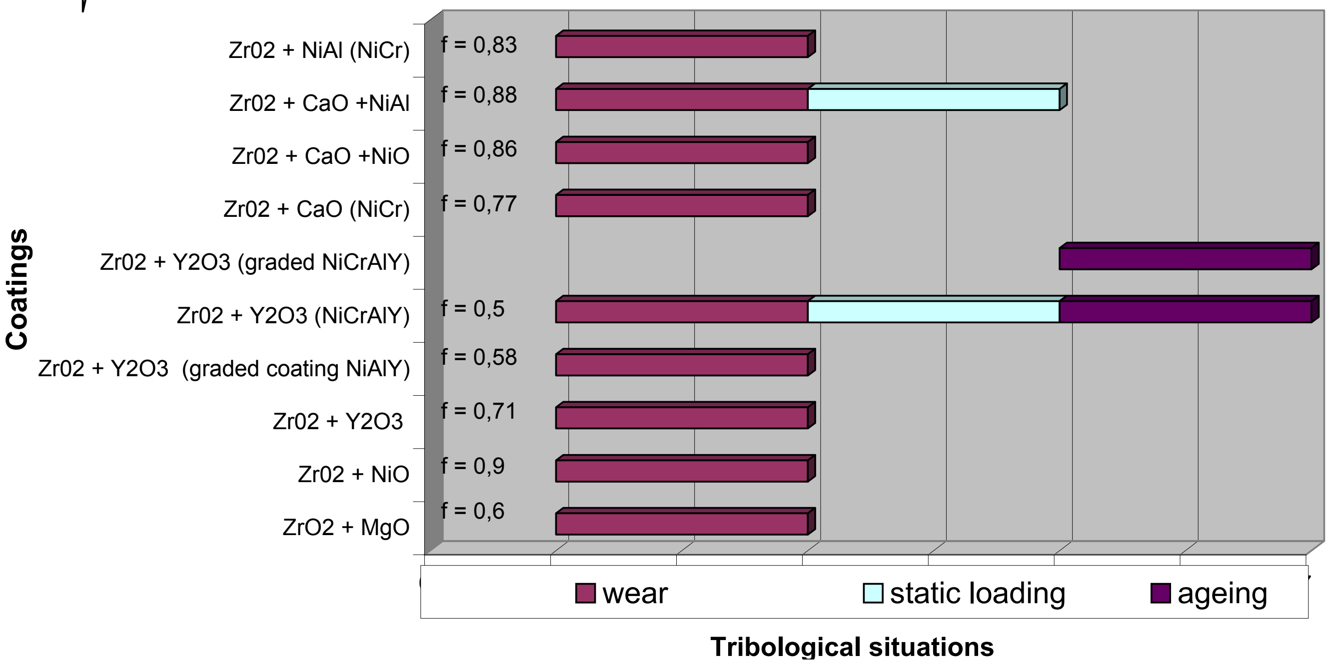


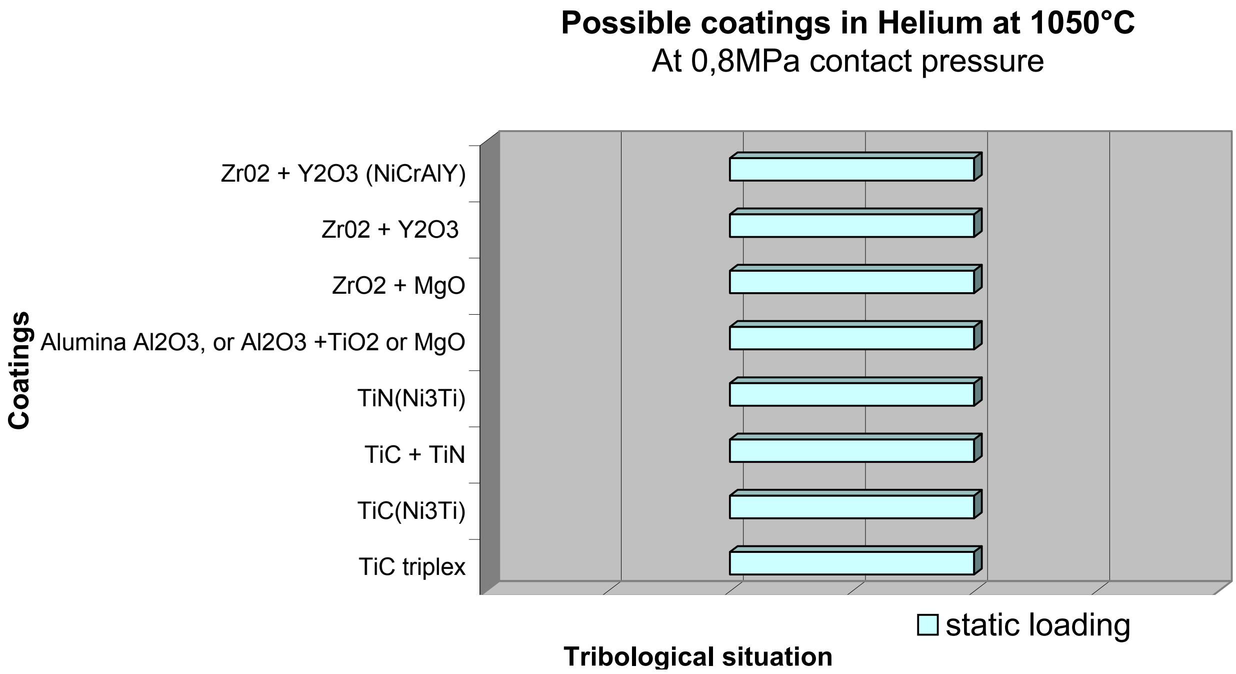
Possible coatings in Helium at 950°C
Contact pressure: 5MPa



ZOOM:

Possible Zirconia-based coatings in Helium at 950°C
Contact pressure: 5MPa





At 1050°C, several coatings used at 850-950°C such as Cr_2C_3, $Cr_{23}C_6$, NbC families are not adapted. Are remaining the $Zr0_2$, TiC/TiN , Al_2O_3 families for static loading applications.

6.4. *Intermediary summary*

At this level of the D39 work, it can be noted that, apart the cobalt based alloys or coatings which are not nuclear compatible, few bare alloys or coatings can be proposed:

- The colmonoy 6 and triballoy 700 are suggested by FZJ in the conclusion
- The Molyddenum based coatings are known for their lubricating properties at 100-600°C (ex of MoS_2 in NNC report D37), but seem to be not adapted to high temperatures: they give bad results at T>850°C (Mo_2C + Mo, TiB_2 + Mo).
- Cr_3C_2 / NiCr coatings close to LC-1B or LC-2 type shown by NNC till 700°C, gave good results in the 600-850°C range, with higher friction coefficient in Helium environment than in CO_2 environment :
  - In CO_2, f in the range of 0.35 at 550-650°C against 316L or LC-1B
  - In Helium, f in the range of 0.4-0.5 at 816°C against Hastelloy X with low wear, and 0.8 - 0.98 against itself in the range 850-950°C

They are retained by FZJ as chromium carbide cermet and duplex in the range respectively of 20-600 and 20-800°C. It has to be noted that the cermet use was restricted by NNC to the 20-200 and 450-700°C, with a 200-450°C zone where the chromium carbide was not recommended in AGR. For turbines, this cermet was also used on a large range of temperatures.

- TiC presents low coefficient of friction at temperatures below 600°C. At higher temperatures, the coefficient increases due probably to softening of the TiC. Nevertheless in its $TiC(Ni_3Ti)$ combination, the coating is suggested at 850 and 950°C.

- ZrO_2 + Y_2O_3 (NiCrAlY or NiAlY) presents friction coefficients close to 0.5 in Helium environment are retained in the FZJ selection whatever the type of coating APS or VPS. They are also specified for thermal barrier.

7. Review of materials and coatings used in other industries (FANP contribution [7])

The specific task of Framatome-ANP was to search data from aerospace, turbines, specific coating.
The temperature of most of the test is between 400 and 800 °C. The motion is essentially sliding, and the tests are conducted under several environments like air, argon or vacuum.
Four papers deal with very high temperatures between 1000°C and 1400°C. The couple that gave the best result was [alumina + MgO + impurities, sintered] / [alumina + MgO + impurities, sintered] with a friction coefficient of 0.18 at 1200°C in air. The couple zirconia (partially stabilized with MgO, sintered) / silicon carbide (Al sintered aid, sintered) had a maximum of friction coefficient, in air, of 0.6 at 600°C that decrease when the temperature increase (0.4 at 1000°C). Other couples have friction coefficient over 0.6 at temperature of 1000°C.
Some other materials have been tested in vacuum. α-SiC, sintered / α-SiC, sintered had a friction coefficient of 0.75 at 1200°C in air and 0.6 at 1200°C in vacuum. 3-D C-C composite / 3-D C-C composite had a friction coefficient between 0.2 and 0.4 at 1000°C in vacuum.
Solid lubricants have been tested too like MoS_2 and WS_2 against 440C stainless steel under argon. The friction coefficient at 1000°C was about 0.3.

Coatings based on ZrO_2 have been tested up to 800°C in air against an alumina ball. Y_2O_3 ZrO_2 alone has a high level of friction coefficient (near 1). Some composites like ZrO_2-$BaCrO_4$ or ZrO_2-CaF_3 have lower friction coefficient at 800°C (between 0.5 and 0.6). The added lubricants improve the friction performance of ZrO_2.

PS300 (a metal bonded chrome oxide coating with Ag and BaF_2/CaF_2 lubricant additives) tested against alumina exhibited lower friction coefficients in air, but the maximum temperature tested was 650°C. At this temperature, the friction coefficient was 0.19 against Al_2O_3.

All this information are listed in the tables 11a to 11g.

A summary is given here after in tables A1 and A2 corresponding respectively to the two following environments: air / oxidising atmosphere and vacuum/inert atmosphere (argon, ...).

In these tables, it appeared that:

- In air, **Zirconia** with MgO or SiO_2 additives are used as well as in helium environment, with close friction coefficient 0.6-0.8 at 1000°C. Lubricant like $BaCrO_4$ was successful in lowering the friction coefficient till 0,5 of zirconia above 300°C. **Chromium oxide Cr_2O_3** were used at 450°C with 0.5-0.4 coefficient of friction, which can be lowered till 0.08-0.09 with a Ca, S, P, Zn lubricants.

Table A1: results in air extract of [7]

Matérial		Temperature	Environment	Application	Results
Part 1	Part 2				
Alumina (99,7%) +MgO + impurities, sintered	Alumina (99,7%) +MgO + impurities, sintered	23-1200°C	air		Increase of friction coefficient at 200°C to 600°C Decrease after 600°C to obtain a value of 0,18 at 1200°C
ZrO2, partially stab. with MgO, or Alumina, + SiO2 or not	Zirconia, partially stabilized with MgO, or Silicon carbide	25-1200°C	air		this tests gave similar results friction coefficient near to 0,2 at room temperature, near 0,6 at 600°C, and 0,7-0,8 at 1000°C
ZrO2, partially stabilized with MgO, sintered	Silicon carbide, Al sintering aid, sintered	23-1200°C	air		friction coefficient less than 0,2 at room temperature, increase to 0,6 at 600°C, and fall to 0,4 at 1000°C
Silicon nitride, gas-pressure sintered	Silicon carbide, Al sintering aid, sintered	23-1400°C	air		friction coefficient at 0,2 at room temperature, increase to just under 0,6 at 600°C, and then to just over 0,6 at 1000°C
Low-pressure plasma-sprayed (LPPS) ZrO_2-$BaCrO_4$ (ZB)	10 mm sintered Al_2O_3 ceramic ball	Room temp to 800°C	laboratory air	for example jet engine	**BaCr04 acts as a lubricant above 300°C -** results at 50N load friction coefficient at 0,8 at room temperature, 1 at 100°C (max value), slightly decreases to 0,92 at 200°C, greatly decreases to 0,68 at 300°C, 0,58 at 400°C, 0,61 at 500°C, 0,57 at 600°C, 0,51 at 700°C and 0,50 at 800°C
8 wt.% Y_2O_3 partially stabilised ZrO_2 (YPSZ)	10 mm sintered Al_2O_3 ceramic ball	Room temp to 800°C	laboratory air		results at 50N load friction coefficient at 0,34 at room temperature, 0,3 at 200°C, increases to 0,63 at 400°C, 1 at 600°C, slightly decreases to 0,83 at 700°C, and increases to 0,97 at 800°C
amorphous hydrogen free carbon (a-C) or with Titanium (Ti-c H)	steel balls of S6-5-2 (AISI M 2) 65 HRC ∅ 4,762 mm	Ambient	In air with humidity variyng from dry to moist (90%)		In air with humidity varying from dry to moist (90%) the coatings showed a rather stable steady-state friction coefficient between 0.06 and 0.21. For all coatings the coefficient decreased with increasing relative humidity >20%. It is noteworthy that the friction coefficient ot Ti-C:H decreased from 0.12 to 0.06 when the relative humidity was lowered from 20% to 1%.
Cr_2O_3	Cr_2O_3	450°C	ambient air	ceramic coatings plasma spraying technology	friction coefficient of 0.52 for 0.08 GPa and 0.47 for 0.2 Gpa. - **friction coefficient of 0.0,08-0,09 when lubricated with a Ca, S, P, Zn based lubricant** Lowest wear depth
Cr_2O_3	TiO_2				friction coefficient of 0.53 for 0.08 GPa and 0.40 for 0.2 Gpa, **and 0,07-0,08 with lubricant**
Cr_2O_3	Al_2O_3				friction coefficient of 0.61 for 0.08 GPa and >0.64 for 0.2 GPa and **0,07-0,08 with lubricant**
Cr_2O_3	Cr3C2				friction coefficient of 0.66-0,48 at 0,08 Gpa and 0,20 GPa load
c-BN coating thickness 0,4 µm, on a silicon wafer (disc)	Stainless steel ring (SUS 304)	400°C	ambient air	c-BN coatings expected to be used as solid lubricants for space applications	in ambiant air friction coefficient is about 0.35; in vacuum at 400°C, the coefficient rapidly reduced to 0.009 at stable stage Friction coefficient 0,7 (high because the properties of SUS 304 changed in these conditions) Adherents (SS) on the c-BN coating surface
10 mm thick alacrite disk with chromium carbide / calcium fluoride bilayer (Cr_3C_2/CaF_2)	Diam. 8 mm aluminia ball average surface roughness 0.03 µm	500°C and 700°C	ambient air	space shuttles HERMES application + bearings for rolling mills (700°C) and dynamic seals at 500-700°C + hot gas	Good results were obtained at 700°C with the **Cr_3C_2 3µm/CaF_2 3µm coating** : static and dynimic friction coefficient respectively 0.25 and 0.28 - High ductility of CaF2 at 700°C limited by Cr3C2 matrix - Lubricant deposited by radiofrequency sputtering.
AISI 304 stainless steel ♦6□ 25 x 7 mm^2 plates coatings ZrO_2-CaF_3 composite coating deposited by LPPS and YPSZ	∅ 10 mm Al_2O_3 ceramic sphere	room temperature to 800°C	laboratory air	plain journal bearings, space shuttles, hot sections of small jet engines	The **LPPS ZrO_2–CaF_2 composite** exhibited a distinct improvement in wear resistance and frictional characteristics as compared to YPSZ coating more porous, less adherent. At RT, the friction coefficient of the ZrO_2–CaF_2 composite decreased with increasing load from 30–80 N. But this situation was reversed for wear loss at RT. At 600 or 700°C, the composite exhibited lower friction and wear than at RT, 400 and 800°C.
PS300 (a 60%chrome oxide 20% NiCr coating with Ag (10%) and BaF_2/CaF_2 lubricant additives (10%))	Al_2O_3 (ceramic) pin 25.4 mm radius of curvature	25°C 500°C 650°C	air		friction coefficient Al2O3 :about 0.62 at 25°C about 0.32 at 500°C about 0.19 at 650°C
	inconel X-750 (metal) pin 25.4 mm radius of curvature				friction coefficient IncX750 about 0.23 at 25°C about 0.29 at 500°C about 0.31 at 650°C

Table A2: results in vacuum or inert atmosphere – extract of [7]

Matérial		Temperature	Environment	Application	Results
Part 1	Part 2				
TiN-coated ring on JIS SUS 304	TiC-coated disc on JIS SUS 304	room to 400°C	vacuum 2,6.10-3 Pa	solid lubricant for space applications	The friction coefficient of the couple decreased to a minimum of 0.2 with the rise of temperature below 350°C and then the friction coefficient increased to 0.38 at 400°C - **same FZJ: deterioration of the oxide film or softening of TiC?**
		400°C	vacuum 1.10-3 Pa		friction coefficient : 0,59
		400°C	vacuum 3,4.10-1 Pa		friction coefficient : 0,78
a-C:H disc	Stainless steel pin	room temperature	vacuum 10-4 Pa argon or helium or molecular hydrogen	tools in contact with non-iron materials	friction coefficient : 0,08
Cubic boron nitride	Stainless Steel	400°C	vacuum 1,7.10-3 Pa		friction coefficient : 0,35 The friction coefficient decrease from an initially high value of 1,1 to 0,009 at steady state. No wear at all was visible.
MoS_2	440C stainless steel	room to 1000°C	argon		about 0.1 under 400°C increase to 0.22 from 400°C to 700°C between 0.2-0.25 above 700°C
WS_2	440C stainless steel	room to 1000°C	argon		about 0.1 under 700°C increase from 0.1 to 0.25-0.3 from 700°C to 800°C about 0.3 above 650°C
sintered silicon carbide α-SiC ring	sintered silicon carbide α-SiC plate	room temperature to 1200°C	vacuum 7-8 Pa		friction coefficient about 0.15 at room temperature rapidly to 0,7 at 400°C, then to 0.85 at 1000°C and to 0,75 at 1200°C in air, and about 0.45 going up to 0.65 at 400°C and to 0.55 at 1000°C in vacuum
sintered silicon carbide α-SiC ring	sintered silicon carbide α-SiC plate	room temperature to 1200°C	vacuum 10-12 Pa		friction coefficient about 0.7 at room temperature, increasing to 0.85 at 800°C and going to 0.75 at 1200°C in air, and about 0.6 going up to 0.65 at 200°C and to 0.6 at 1200°C (nearly constant) in vacuum
c-BN coating thickness 0,4 µm, on a silicon wafer (disc)	Stainless steel ring (SUS 304)	400°C	$1{,}7.10^{-3}$ Pa	c-BN coatings are expected to be used as solid lubricants for space applications	in ambiant air friction coefficient is about 0.35; in vacuum at 400°C, the coefficient rapidly reduced to 0.009 at stable stage
		600°C	vacuum $1{,}3.10^{-3}$ Pa		Friction coefficient 0,7 (high because the properties of SUS 304 changed in these conditions) Adherents (SS) on the c-BN coating surface
Si3N4 pin diam. 12.7 mm	ceramics : SiC, TiC, ZrO2, Al2O3 and Si3N4	room temperature,200°C, 400°C and 600°C	nitrogen gas atmosphere compared to vacuum		high friction coefficient (about 1) decreasing with the presence of wear particle between surfaces in sliding contact. Wear rate at 600°C is lower in nitrogen gas than in vacuum, except for Si3N4 and SiC disks.

A 60% Chromium oxide with 20%NiCr and 20% (Ag + BaF_2/CaF_2) was also used with success (f=0.19 on Al_2O_3 and 0.31 on inconel X750 at 650°C). **Chromium carbide** Cr_3C_2 was used with a CaF_2 lubricant (f= 0.25-0.28). Carbon based coatings seem to be interested to study combined with Titanium (Ti-c-H), Si (like SiC) or BN (as c-BN) since in these cases, friction coefficients are quite low.

- In vacuum or inert atmosphere such as argon, lubricant coatings such as **TiN** at 400°C, WS_2 and **MoS_2** with Stainless Steel, **c-BN** coatings on S.S., are proposed. **Si_3N_4** can be used against ceramics such as ZrO_2, Al_2O_3, in nitrogen atmosphere.

8. Conclusion: recommendations for materials and coatings

A number of materials such as Stainless Steel 316, other austenitic steels type 347, alloy 800, carbon steel, 9Cr steel, or nickel alloys and graphite, are envisaged in PBMR and GTMHR for several main components such as control rod drive mechanisms, hot gas duct, stators seals, internals…
No coating was proposed in the past experience, although they are mainly austenitic steels, that suffer from poor tribological performance.

The temperature ranges are:
- 100-500°C
- 500-800°C
- upper than 800°C

The present study has shown the following results from past experience:

- At lower temperatures (100-500°C) in Helium environment:

Bibliography showed some examples of metallic couples presenting grooves or scratches and material transfer due to bad tribological properties: from instance Hastelloy X like-to-like at 20°C, In713LC / X20CrMoV12-1 couple at 350°C or In713LC / 17CrMo5-5 at 480°C.

- In the 500-800°C range

In Helium atmosphere, several metallic combinations based on Stainless Steel, mild and low alloy steel, Nimonic alloys, Incoloy 800, …showed more or less strong adhesion versus temperature (except Nimonic 90 against S.S alloy 321).

- At high temperatures (800-1200°C)

In Helium atmosphere, several metallic combinations based on Hastelloy X, Inconel 601, 625 or Incoloy 800 showed severe galling in absence of any coating or lubricant, at 800 and 1000°C.
Many of these configurations corresponded in the past to real component in AGR, and mostly Dragon or German HTR Module.

Solutions were found by using coatings just as carbide based cermets, or lubricants as MoS_2 cermet to limit wear and rubbing in CO_2 (for AGR) or Helium atmosphere (Dragon, German module). Same or other types of coatings were found in turbine or aerospace industry, corresponding to oxidising atmospheres.

A recapitulative table (**table B**) is given here after, which marks out the possible coatings which are working in what environment and in what range of temperature.

To answer the previous question for PBMR and GTMHR working in helium environment, the following coatings can be suggested and need probably to be tested in specific associated configuration (atmosphere, contact pressure, …):

- ***At lower temperatures (100-500°C) in Helium environment:***
  - Cermet WC_3/Cr_2C_3/NiCr, used particularly on S.S was chosen for low temperatures in Helium environment (although Cermet Cr_2C_3 / NiCr is better for high temperatures). Cermet WC_3/Cr_2C_3/NiCr worked at 20 to 350/400°C.
  - Ni based alloy such as Triballoy, In 713LC like to like, were working in the 200 to 400-500°C range. An other Ni alloy named alloy C(Ni base, 17Cr, 17Mo) was working in CO_2 atmosphere in the range 350 to 500°C. It would had to be verified in Helium environment with some oxidising species.
  - Lubricants such as MoS_2 cermet, WS, cubic boron nitride (BN), TiN or nitriding process were efficient against S.S to lower wear in vaccum or argon environment. It would be interesting to test their efficiency in Helium.
  - Zirconia / NiCrAlY or Alumina, stabilised with various components such as CaO, NiO, MgO, were used at these temperatures, but in oxidising environments.

- ***In the 500-800°C range***
  - Concerning Nickel alloys and couples like-to-like with Inconel 625, Triballoy, Colmonoy 6, good results were obtained till 650°C
  - Cermet Cr_2C_3 / NiCr and cermet WC_3 / Cr_2C_3 / NiCr were quite good for dwelling, against adhesion and have a good oxidation behaviour, whatever the mode of deposition D-Gun or plasma gun, except on Hastelloy X.
  - Same for Zirconia / NiCrAlY or Alumina, stabilised, but used in oxidised environment. To be verified in helium atmosphere under 800°C.
  - Lubricants such as MoS_2 cermet and WS, were still efficient against S.S in this 500-800°C range.

- ***At high temperatures (800-1200°C)***

  - Cermet Cr_2C_3 / NiCr till 950°C (also for fretting) and $Cr_{23}C_6$;
  - Zirconia / NiCrAlY or Alumina based coating were well adapted in helium environment, and even in oxidising atmosphere;
  - Other coatings such as TiC(Ni_3Ti), SiC and NbC(NiCr) seemed to be quite interesting to be used in Helium environment in 850-950°C range. Hastelloy X coated with TiN was working at 800-900°C;
  - Lubricants such as MoS_2 cermet and WS, were still efficient against S.S in the 800-1000°C range.

In any cases, it is necessary to keep in mind the following points:

- The friction coefficient is important, but the wear too;
- The result is function of the solicitation type (sliding, fretting, dwell, …) and of the environment (quantity of species);
- Considering the environment, the concentration of water and O_2 had considerable effect on the friction coefficient and on the wear. In future tests, normal and abnormal conditions (correct levels of impurities or not) should be considered.
- For the coatings, it is necessary to take care of the coating process and the capability to coat the surface (dimension, shape, internal hole, roughness, …);
- Wear and friction occur on a material couple, so the two counter parts have to be mentioned;
- We have to keep a close eye on new materials and new deposition processes (for example for SiC), that could be tested in HTR environment.

Table B: Coatings and materials versus temperature and gaseous environment – SLIDING APPLICATION

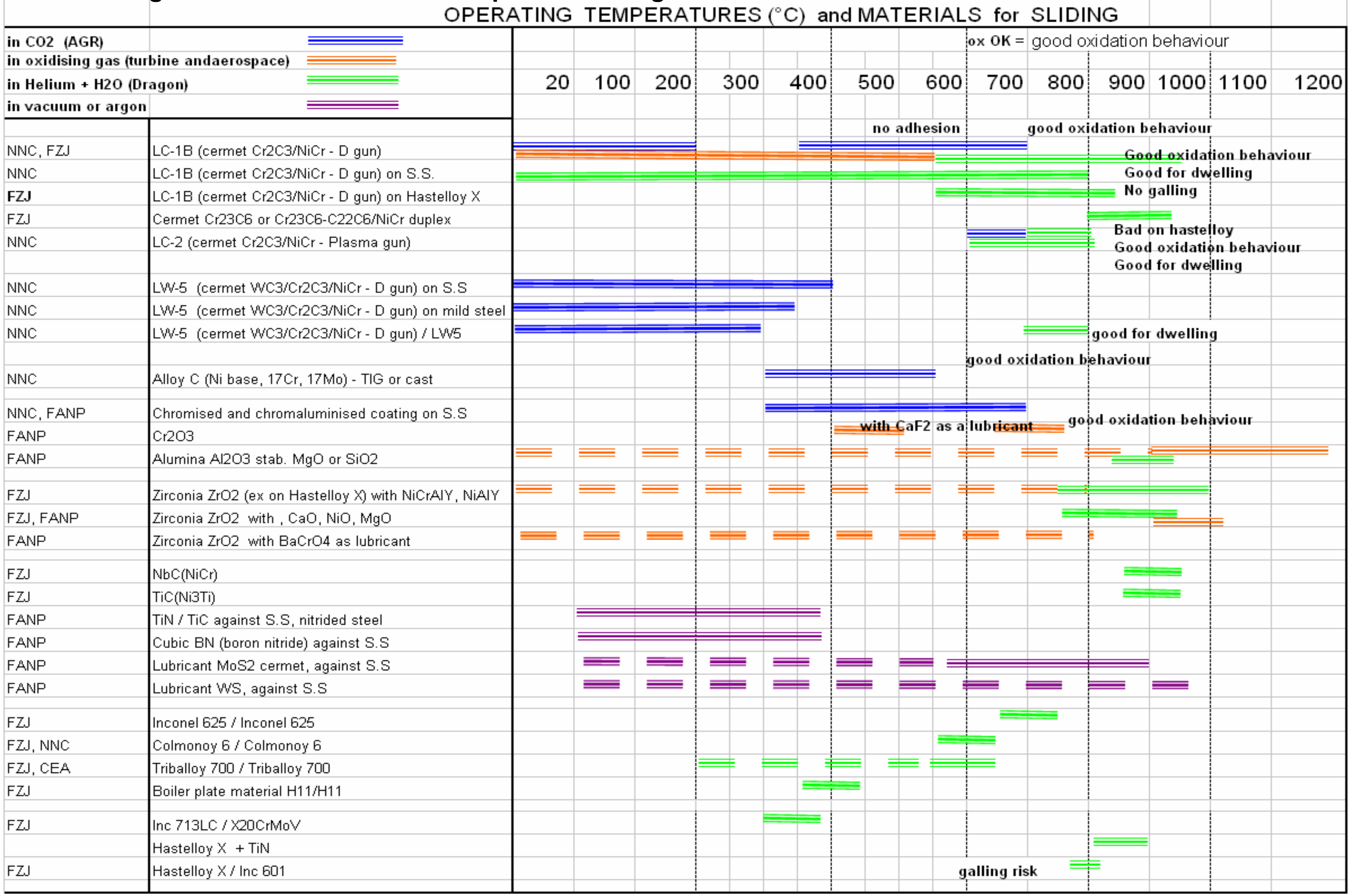
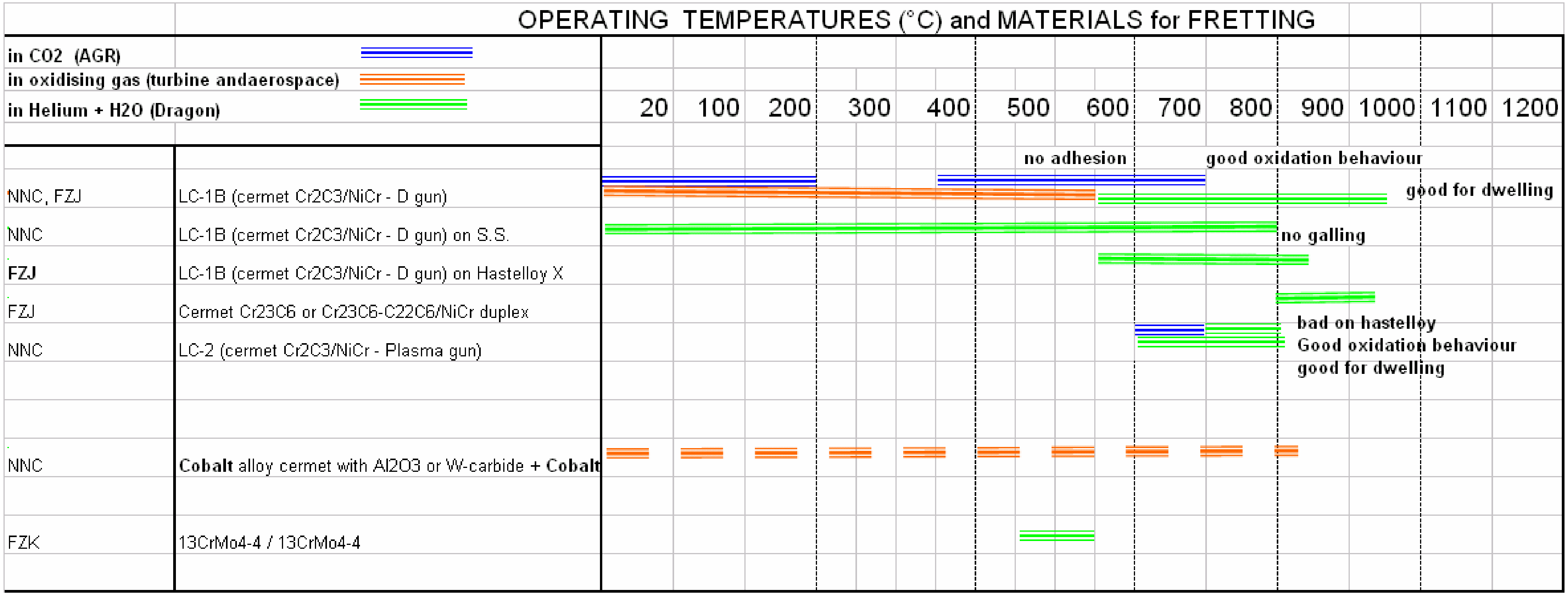


Table C: Coatings and materials versus temperature and gaseous environment – FRETTING APPLICATION



LIST OF TABLES

EXTRACTED FROM PARTNERS CONTRI-BUTIONS

Table 1: possible material problems in PBMR according to several components [3]

Component	Area	Temperature	Type of Motion	Possible Materials	Potential Problems
Helium Inventory Control System	valves compressors				static adhesion wear and leakage
Heat Exchangers	pre-cooler inter-cooler	<150°C	thermal movement vibration	carbon steel	fretting adhesive wear rubbing
Power Turbine	magnetic bearings braking system	700°C			
Recuperator	plate supports	500- 700°C		austenitic steel (type 347)	
Turbo Units (LP & HP)	bearings (electromagnetic. axial, capture)		sliding		high speed, long life fretting erosion due to dust
Reserve Shut Down System	storage container valve		sliding		static adhesion wear and leakage
Reactivity Control System (Control Rod)	drive mechanism	450 - 900°C	sliding motion	alloy 800 or similar	wear gear wear chain drive wear contact adhesion
Core	pebbles blocks bricks keys supports steel barrel	450 - 900°C	thermal movement sliding	graphite type 316 stainless steel	brick restraint brick movements thermal cycle wear cracking from keyed slots
Core Conditioning System	valves blowers service hatch heat exchanger	~150°C	sliding		static adhesion wear and leakage

Table 2: Materials used in the AGR reactor at T ≥ 300°C - extract of [4]

Main components	Material for parts of these components	Operating temperature (°C)	Example of coating
Primary economiser (boiler tubes)	Mild steel, 1Cr	≤ 320-345°C	
Control rod joints	Austenitic Steel Cr-Ni (316, 321)	430°C	LC-1B
Secondary economiser, evaporator, primary superheater (boiler tubes)	9Cr	≤ 530°C	
Boiler unit	Austenitic Steel Cr-Ni (316, 321)	564-589°C	Alloy C welded or cast
Superheater	Austenitic Steel Cr-Ni (316, 321, SA194), Nimonic (Ni,Cr)	589°C	
Reheater unit	Austenitic Steel Cr-Ni (316, 321)	589-610°C	
Secondary superheater, reheater (boiler tubes)	Austenitic Steel Cr-Ni (316)	600°C	
Guide tubes	Austenitic Steel Cr-Ni (316, 321, WHB24)	400-675°C	LC-2
Gag unit	Austenitic Steel Cr-Ni (316, 321,347)	650°C	LC-1B – Alloy C

Table 3 Compositions of hard coatings used in UK AGRs [4]

Coating	Manufacturer	Coating type	Deposition technique	Composition (%)
LC-1B	Union Carbide (now Praxair)	Cermet	D-Gun	80 vol Cr_2C_3, 20 vol NiCr
LC-2	Union Carbide (now Praxair)	Cermet	Plasma Gun	75 vol Cr_2C_3, 25 vol NiCr
LW-5	Union Carbide (now Praxair)	Cermet	D-Gun	25 WC, 70 mixed WC_3/Cr_2C_3, 5 Ni
Alloy C	Deloro Stellite	Metallic	TIG weld or cast	Ni base, 17 Cr, 17 Mo, 6 Fe, 5 W, 0.1 C
Alloy 50	Deloro Stellite	Metallic	TIG weld, cast or spray fused (SF50)	Ni base, 10 Cr, 4 Fe, 4 Si, 1.8 B, 0.5 Cu, 0.4 C
Alloy 60	Deloro Stellite	Metallic	TIG weld, cast or spray fused (SF60)	Ni base, 16 Cr, 4.5 Fe, 4.5 Si, 3.5 B, 0.5 C

Table 4: AGR coatings operating at temperatures above 300°C up to 750°C - [4]

Coating	Base material	Max temp (°C)	Component
LC-1B	-	400	Thermal baffle
	En 58E or F	560	Control rod joint
	-	600	Control rod joints
	BS 1631B (austenitic CrNi)	650	Gag orifice, guide bearing, universal joint
	316	650	Gag plug fins
	BS 1631B (austenitic CrNi)	650	Gag orifice
	321	650	Gag drive shaft
	316	650	Fuel assembly
	304	650	Plug unit assembly, gimbal pin and bush
LW-5	321	300	Seal in flux scan penetration
	Mild steel, 316	300	Gag and circulator items, gas bypass bellows
	321	320	SSD tail pipe
	BS 1631B (austenitic CrNi)	400	I/S guide tube
	304	400	I/S guide tube adaptor sleeve
LC-2	-	650	Fuelling and controlling rod guide tube
SF50	-	650	Gag drive shaft
Alloy 50	-	370	Core restraint
SF60	304	300	SSD
	18/8	725	Fuel stringer – I.C. tube insert
Alloy C	Mild steel	300	Gag shaft
	-	600	Boiler seals
	-	640	Upper tail pipes
	316	650	Gimbal joint
	-	650	Boiler hangers
	321	750	Control rod
PW-60	304	350	SSD pipework support
Chromised	Mild steel	438	
	316	630	S/H tail tube grid support

Table 5A: Coatings used in UK AGR reactor for temperatures up to 400°C - [4]

Component	Coating	Coating thick-ness (µm)	Counterface	Action	Temp (°C)
Top bush retaining ring – gas circulator	LW-5				280
Dome guide – gas circulator (or stop ring)	LW-5				280
Isolating dome - gas circulator	LW-5				280
Seal ring (piston ring) – gas circulator	LW-5				280
Inner sealing ring – gas circulator	LW-5				280
Bottom bush – gas circulator	LW-5				280
Dome operating rods – gas circulator	LW-5				280
Motor piston ring - gas circulator	LW-5	50-75	LW-5	Sliding	280
Inner seal ring-circulator dome transfer ring	LW-5	50-75	Mild steel	Sliding	280
Transfer ring bore-circulator dome	LW-5	75-100	LW-5	Sliding	280
Dome operating rods - gas circulator	LW-5	175-225	Nitrided EN40B (3 Cr-Mo)	Sliding	280
Hot box labyrinth seal	LW-5				280
Flux scan penetration – gas sample pipe bearing seal	LW-5		EN58B (17/20 Cr, 7/10 Ni)		300
Gag shaft vibration snubber and rubbing button	Alloy C	50-100	Mild steel	Sliding/fretting	300
Core restraint	Stellite Alloy 50				350
Dome thermocouple penetration – thermal baf-fle:segmented ring	LC-1B		EN58B (17/20 Cr, 7/10 Ni)		300-400
Interstitial guide tube	LW-5	50 mini	BS 3100 347C17 (18/21 Cr, 9/12 Ni) casting	Sliding	400

Table 5B: Coatings used in UK AGR reactor for temperatures up to 725°C [4]

Component	Coating	Coating thick-ness (µm)	Counterface	Action	Temp (°C)
Boiler thermocouple duct liners	'High Cr' Alloy C welded		'High Cr' Alloy C cast	Adhesion	290-640
Boiler hangers – anchors and guides	Alloy C			Rubbing/adhesion	280-650
Control rod shock absorber – piston in swage tube	Alloy C			Rubbing	350-750
Boiler interbank seals	Alloy C welded		Alloy C cast	Sliding	500 & 600
Control rod joint	LC-1B	50 mini	LC-1B	Rocking	560
Control rod joints	LC-1B		LC-1B		600 maxi
Boiler spectacle plates on upper tail pipes	Alloy C cast			Bearing location	640
Fuelling guide tube top bearing (hub and housing)	LC-2		LC-2		650
Gag orifice	LC-1B	50 mini	LC-1B	Sliding/fretting	650
Gag plug fins	LC-1B	50 mini	LC-1B	Sliding/fretting	650
Gag drive shaft-Hooke joint-pin	Alloy C	50-100	Alloy C	Sliding	650
Gag drive shaft guide bearing	LC-1B	50 mini	EN 58B (17/20 Cr, 7/10 Ni)	Sliding/fretting	650
Fuel assembly-sealed gimbal joint-pivot pins	Alloy C	150 mini	BS 3100 347C17 (18/21 Cr, 9/12 Ni) casting	Sliding	650
Fuel assembly - gag coupling universal joint	LC-1B	75-100	BS 3100 347C17 (18/21 Cr, 9/12 Ni) casting	Sliding/fretting	650
Fuel stringer – IC tube insert	Stellite SF-60			Rubbing	725

Table 6 Potential HTR problems identified during design, construction and operation of Dragon (from NNC report) – [D37 – [5]]

Component	Potential problem	Temperature range (°C)	Type of motion	Possible materials	Comments
Insulation foil	(a) fretting due to noise (b) movements on thermal cycling	350-800	(a) high frequency, low amplitude (b) low frequency	18/8/1 20/25/Nb	No problem found at Dragon
Boiler tubes	Fretting due to noise. Wear due to thermal movement.	350-800	High and low frequency. High and low amplitudes.	Low temp: mild steel Medium temp: 2¼Cr/Mo, 9Cr High temp:18/8/1, 20/25/Nb, Incoloy	Slight wear due to thermal movement found in Dragon primary heat exchanger
Channel gags	Bearings at outlet temperature	600-850	Occasional sliding and/or rotation	Hard facing, graphite and MoS_2, cermets and ceramics	Need for gag adjustment depends on detailed design
Channel seals	Effects at high temperatures	400-850	Insertion and thermal movements	Graphite and MoS_2,	Friction coefficient > 0.4 could give fault condition
Channel gag drives	Drying out of greases. High temp bearings	100-600	Sliding and/or rotation	Conventional materials + grease graphite + MoS_2 hard facings on lubricated metals. Dry lubricated metals.	Maximum temp/life of greases to be assessed.
Thermal expansion joints	Surfaces moving due to thermal movements	Ambient -800	Sliding intermittently	Mild steel - Nimonics	Iron piston rings on Nimonics used on Dragon
Articulated control rods	Wear and seizing	350-550	Rolling/sliding	18/8/1 Nimonics, 9Cr Nitrided low alloy steel	No problem on Dragon.

Table 7: Results of tests in Dragon: [4]

Tests type 1: in pure helium at 0.1 MPa or 5 MPa pressure,

Tests type 2: in impure (reactor) helium at 2 MPa, at temperatures from 20°C to 800°C. The partial pressures of the impurities in the reactor helium were as follows. H_2O: 5 Pa, H_2: 50 Pa and CO: 50

Material 1	***Material 2***	***Temperature***	***Pure helium (test 1)***	***Impure Helium (test 2)***
LC-1B (chromium carbide)	LC-1B (chromium carbide)	20-600°C	This couple wore severely at 20°C in both atmospheres. With increasing temperature the wear rate fell;	
LC-1B (chromium carbide)	LC-1B (chromium carbide)	600-800°C	at the higher temperatures it was very low. Friction coefficients were moderate, in the range 0.5 - 1.0.	
Alloy C	Type 316 S.S.	20-800°C	The performance in all tests was poor. The lowest friction coefficient observed was 1.0. In most tests there was severe galling.	
Tungsten carbide (LW-5)	Tungsten carbide (LW-5)	20-800°C	at 0.1 MPa (gas) and 6.5 MPa (contact) friction coefficients around 0.5 at 20°C and 800°C severe wear at the lower temp. but very low wear at 800°C.	-
Chromium carbide (LC-2)	Chromium carbide (LC-2)	20-800°C	Id. + after the tests at 800°C, the LC-2 specimens adhered.	
Stellite 6	Stellite 6	800°C	Galling at high temp.	
Chromium carbide (LC-1B)	Type 316 S.S.	20- 800°C	unsatisfactory at all temperatures in pure helium, galling badly.	

Pa.

Table 8: Results of tests in Dragon: [4]

Friction & Wear: - Dwell Tests - in-reactor conditions , 450-750°C, 100 cycles /day & 10 cycles/day – 20 days

		Temperature	Dwell test	Continuous test
Alloy C	AISI 316	-	More severe wear for dwell test	No unambiguous difference in friction coefficient between the dwell tests and the continuous tests.
AISI 316	AISI 316	650 and 750°C		
Nimonic 90	AISI 316	650 and 750°C		
Nimonic 90	Nimonic 90	650 and 750°C		
Chromium carbide LC-1B	Chromium carbide LC-1B	650 and 750°C	No effect of dwell on load	
AISI 316	EYC9106 Graphite	450-750°C	No effect of dwell on load	
En40B	En40B	450°C		

Table 9 Coatings applied to gas turbine components in the compressor section, T < 1000°F (538°C) [4]

Components	Coated area	Tribological problem	Coating type
Fan and compressor blades	Shroud pads	Fretting	Tungsten carbide/cobalt
Fan and compressor blades	Root section pressure faces	Galling	Copper-nickel-indium
Variable vane trunions, drive arms, bearings, etc	Bearing surfaces	Fretting	Tungsten carbide/cobalt
Compressor hubs	Bearing journal diameters	Fretting	Tungsten carbide/cobalt
Compressor blades	Airfoils	Particle erosion	Tungsten-titanium carbide/nickel, tungsten carbide/cobalt, titanium nitride
Compressor hubs and discs	Snap diameters	Fretting	Tungsten carbide/cobalt
Expansion joints	Sealing surfaces	Sliding and fretting	Tungsten carbide/cobalt
Diffusers and impellors	Vane surfaces	Particle erosion	Tungsten-titanium carbide/nickel, tungsten carbide/cobalt, titanium nitride
Bearings	Sealing surfaces	Sliding	Chromium carbide/nickel-chromium, tungsten carbide/cobalt,
Bearings	Sealing surfaces	Fretting	Tungsten carbide/cobalt
Gears	Bearing surfaces and journals	Fretting	Tungsten carbide/cobalt
Labyrinth seal fins	Knife edge tips and face	Rubbing	Tungsten carbide/cobalt, chromium carbide/nickel-chromium, aluminium oxide

Table 10 Coatings applied to gas turbine components in the combustion and turbine sections, T > 1000°F (538°C) - [4]

Components	Coated area	Tribological problem	Coating type
Combustion chamber positioning pins and bushes	Bearing surfaces	Fretting	Tungsten carbide/cobalt, chromium carbide/nickel-chromium
Fuel nozzle and swirler	Bearing surfaces	Fretting	Chromium carbide/nickel-chromium
Fuel nozzle and swirler	Threads	Loosening	Copper-nickel-indium
Combustion chamber	Interior surfaces	Thermal barrier required	Duplex McrAlY/partially stabilised zirconia
Turbine blades	Unshrouded tip	Rubbing	Cobalt alloy cermet (with alumina dispersion)
Outer airseals	Sealing surfaces	Rubbing	Cobalt alloy
Outer airseals	Sealing surfaces	Thermal barrier required with erosion resistance	Duplex McrAlY/partially stabilised zirconia
Turbine stator shrouds	Shroud flanges	Fretting	Chromium carbide/nickel-chromium, cobalt alloy cermet (with alumina dispersion)
Turbine vanes	Inner-foot pads	Fretting	Chromium carbide/nickel-chromium, cobalt alloy cermet (with alumina dispersion)
Exhaust fairing pins and bushes	Bearing surfaces	Fretting	Tungsten carbide/cobalt

Table 11a: data obtained from turbine and aerospace industry - [7]

Publication	Matérial Part 1	Part 2	Motion	Temperature	Environment	Application	Results
1	INC 738 LC	SiSiC	Oscillating sliding	Room temp to 700°C	air		Influence of the grain size and quality at high temp. levels. Increase of wear ~400-500°C Decrease after for grain-refined modification Transition : transfer of metal on the ceramic surface
2	γ-TiAl Ti-48Al-2Cr-2Nb	Nickelbase superalloy	fretting	23-550°C	air	Fan and compresssor blades Fretting in fit interfaces at the dovetail.	Increase of wear at 200°C Wear (with & frequency
3	Alumina (99,7%) +MgO + impurities, sintered	Alumina (99,7%) +MgO + impurities, sintered	sliding 0,24 mm/s, stroke of 2 mm	23-1200°C	air		Increase of friction coefficient at 200°C to 600°C Decrease after 600°C to obtain a value of 0,18 at 1200°C
	Zirconia, partially stabilized with MgO, sintered	Zirconia, partially stabilized with MgO, sintered	sliding 0,24 mm/s, stroke of 2 mm	25-1200°C	air		this tests gave similar results friction coefficient near to 0,2 at room temperature, near 0,6 at 600°C, and 0,7-0,8 at 1000°C
	Alumina (99,5%) + MgO + SiO2 + impurities, sintered	Zirconia, partially stabilized with MgO, sintered	sliding 0,24 mm/s, stroke of 2 mm	27-1000°C	air		
	Alumina (99,5%) + MgO + SiO2 + impurities, sintered	Silicon carbide, Al sintering aid, sintered	sliding 0,24 mm/s, stroke of 2 mm	27-1300°C	air		
	Zirconia, partially stabilized with MgO, sintered	Silicon carbide, Al sintering aid, sintered	sliding 0,24 mm/s, stroke of 2 mm	23-1200°C	air		friction coefficient less than 0,2 at room temperature, increase to 0,6 at 600°C, and fall to 0,4 at 1000°C
	Silicon carbide, Al sintering aid, sintered	Silicon carbide, Al sintering aid, sintered	sliding 0,24 mm/s, stroke of 2 mm	26-1400°C	air		high and increasing friction coefficient

Table 11b: data from turbine and aerospace industry (following) - [7]

Publication	Matérial Part 1	Matérial Part 2	Motion	Temperature	Environment	Application	Results
3	Sialon, composition proprietry, sintered	Silicon carbide, Al sintering aid, sintered	sliding 0,24 mm/s, stroke of 2 mm	22-1300°C	air		friction coefficient at 0,2 at room temperature, increase sharply to 0,8 at 600°C, and fall to 0,6 at 1000°C
3	Silicon nitride, composition proprietry, gas-pressure sintered	Silicon carbide, Al sintering aid, sintered	sliding 0,24 mm/s, stroke of 2 mm	23-1400°C	air		friction coefficient at 0,2 at room temperature, increase to just under 0,6 at 600°C, and then to just over 0,6 at 1000°C
4	Low-pressure plasma-sprayed (LPPS) ZrO_2-$BaCrO_4$ (ZB)	10 mm sintered Al_2O_3 ceramic ball	30-80 N load, 10 Hz, stroke 1 mm, duration 1 h	Room temp to 800°C	laboratory air	for example jet engine	results at 50N load friction coefficient at 0,8 at room temperature, 1 at 100°C (max value), slightly decreases to 0,92 at 200°C, greatly decreases to 0,68 at 300°C, 0,58 at 400°C, 0,61 at 500°C, 0,57 at 600°C, 0,51 at 700°C and 0,50 at 800°C
4	8 wt.% Y_2O_3 partially stabilised ZrO_2 (YPSZ)	10 mm sintered Al_2O_3 ceramic ball	30-80 N load, 10 Hz, stroke 1 mm, duration 1 h	Room temp to 800°C	laboratory air		results at 50N load friction coefficient at 0,34 at room temperature, 0,3 at 200°C, increases to 0,63 at 400°C, 1 at 600°C, slightly decreases to 0,83 at 700°C, and increases to 0,97 at 800°C
5	ZrO_2-Y_2O_3 LPPS (low-pressure plasma-spraying)	10 mm sintered Al_2O_3 ceramic ball	30-50 N load, 10 Hz, stroke 1 mm, duration 1 h	Room temp to 800°C	laboratory air		results at 50N load friction coefficient at 0,34 at room temperature, 0,3 at 200°C, increases to 0,63 at 400°C, 1 at 600°C, slightly decreases to 0,83 at 700°C, and increases to 0,97 at 800°C
5	ZrO_2-Y_2O_3 LPHS (laser-assisted plasma hybrid spraying)	10 mm sintered Al_2O_3 ceramic ball	30-50 N load, 10 Hz, stroke 1 mm, duration 1 h	Room temp to 800°C	laboratory air		results at 50N load friction coefficient at 0,23 at room temperature, increases to 0,45 at 400°C, 1 at 600°C, and increases to 1,2 at 800°C

Table 11c: data from turbine and aerospace industry (following) - [7]

Publication	Matérial Part 1	Part 2	Motion	Temperature	Environment	Application	Results
6	sputtered amorphous hydrogen free carbon (a-C)	steel balls of S6-5-2 (AISI M 2) 65 HRC ∅ 4,762 mm	10 N 0.1m/s distance of 1000 m	Ambient	In air with humidity variyng from dry to moist (90%)		In air with humidity variyng from dry to moist (90%) the coatings showed a rather stable steady-state friction coefficient between 0.06 and 0.21. For all coatings the coefficient decreased with increasing relative humidity >20%. It is noteworthy that the friction coefficient ot Ti-C:H decreased from 0.12 to 0.06 when the relative humidity was lowered from 20% to 1%.
	plasma deposited hydrogen containing amorphous carbon (a-C:H)	steel balls of S6-5-2 (AISI M 2) 65 HRC ∅ 4,762 mm	10 N 0.1m/s distance of 1000 m	Ambient	In air with humidity variyng from dry to moist (90%)		
	titanium doped hydrogen containing amorphous carbon (Ti-c:H)	steel balls of S6-5-2 (AISI M 2) 65 HRC ∅ 4,762 mm	10 N 0.1m/s distance of 1000 m	Ambient	In air with humidity variyng from dry to moist (90%)		
	sputtered amorphous hydrogen free carbon (a-C)	steel balls of S6-5-2 (AISI M 2) 65 HRC ∅ 4,762 mm	10 N 0.1m/s distance of 1000 m	450°C	vacuum 3.10^{-3} Pa		Without heating : for all coatings the friction coefficient increased rapidly from values of 0.02-0.08 up to 0.6-0.7 and all the coatings failed within 0.5 m sliding distance. With heating to 450°C, the tribological behaviour was comparable. The coatings were again rapidly damaged within a very short sliding distance because of a sharp increase of the friction coefficient.
	plasma deposited hydrogen containing amorphous carbon (a-C:H)	steel balls of S6-5-2 (AISI M 2) 65 HRC ∅ 4,762 mm	10 N 0.1m/s distance of 1000 m	450°C	vacuum 3.10^{-3} Pa		
	titanium doped hydrogen containing amorphous carbon (Ti-c:H)	steel balls of S6-5-2 (AISI M 2) 65 HRC ∅ 4,762 mm	10 N 0.1m/s distance of 1000 m	450°C	vacuum 3.10^{-3} Pa		
7	Si_3N_4 pin (ball) ∅ 12.7 mm	SiC TiC ZrO_2 Al_2O_3 Si_3N_4	6.02 N oscillating speed 0.2 m/s sliding distance 100-3000 m	25 ; 200 ; 400 ; 600°C	vacuum $5{,}3.10^{-3}$ Pa		The mean friction coefficient was in the range of 0.19 to 0.62 under mild wear and in the range of 0.68 to 1.0 under severe wear and the wear rate of pin increased with an increase in the mean friction coefficient

Table 11d: data from turbine and aerospace industry (following) - [7]

Publication	Matérial Part 1	Matérial Part 2	Motion	Temperature	Environment	Application	Results
8	TiN-coated ring on JIS SUS 304	TiC-coated disc on JIS SUS 304	9,8 N 50 rpm	room to 400°C	vacuum $2,6.10^{-3}$ Pa	solid lubricant for space applications	The friction coefficient of the couple decreased to a minimum of 0.2 with the rise of temperature below 350°C and then the friction coefficient increased to 0.38 at 400°C
				400°C	vacuum 1.10^{-3} Pa		friction coefficient : 0,59
				400°C	vacuum $3,4.10^{-1}$ Pa		friction coefficient : 0,78
9	a-C:H disc	Stainless steel pin	2 N 0,03-0,09 m/s	room temperature	vacuum 10^{-4} Pa argon or helium or molecular hydrogen	tools in contact with non-iron materials	friction coefficient : 0,08
	Cubic boron nitride	Stainless Steel			air		friction coefficient : 0,35
				400°C	vacuum $1,7.10^{-3}$ Pa		The friction coefficient decrease from an initially high value of 1,1 to 0,009 at steady state. No wear at all was visible.
10	Cr_2O_3	Cr_2O_3	10 mn at 0.08 GPa and 20 mn at 0,2 GPa roller: 3,8 m/s block: 1 mm/s, stroke 10 mm	450°C	ambient air	ceramic coatings plasma spraying technology	friction coefficient of 0.52 for 0.08 GPa and 0.47 for 0.2 Gpa. Lowest wear depth
	Cr_2O_3	TiO_2					friction coefficient of 0.53 for 0.08 GPa and 0.40 for 0.2 GPa
	Cr_2O_3	Al_2O_3					friction coefficient of 0.61 for 0.08 GPa and >0.64 for 0.2 GPa (seizure occurred when a 0,2 MPa Hertz pressure was applied for 3 min)
11	MoS_2	440C stainless steel		room to 1000°C	argon		about 0.1 under 400°C increase to 0.22 from 400°C to 700°C between 0.2-0.25 above 700°C
	WS_2	440C stainless steel		room to 1000°C	argon		about 0.1 under 700°C increase from 0.1 to 0.25-0.3 from 700°C to 800°C about 0.3 above 650°C

Table 11e: data from turbine and aerospace industry (following) - [7]

Publication	Matérial Part 1	Matérial Part 2	Motion	Temperature	Environment	Application	Results
12	sintered silicon carbide α-SiC ring	sintered silicon carbide α-SiC plate	0.4 MPa sliding speed 0.2 m/s dist. 500 m	room temperature to 1200°C	air vacuum 7-8 Pa		friction coefficient about 0.15 at room temperature ↗ rapidely to 0,7 at 400°C, then to 0.85 at 1000°C and ↘ to 0,75 at 1200°C in air, and about 0.45 going up to 0.65 at 400°C and to 0.55 at 1000°C in vacuum
	sintered silicon carbide α-SiC ring	sintered silicon carbide α-SiC plate	1.2 MPa sliding speed 0.2 m/s dist. 500 m	room temperature to 1200°C	air vacuum 10-12 Pa		friction coefficient about 0.7 at room temperature, increasing to 0.85 at 800°C and going to 0.75 at 1200°C in air, and about 0.6 going up to 0.65 at 200°C and to 0.6 at 1200°C (nearly constant) in vacuum
13	$12x15x1.6\ mm^3$ flat specimen 3-D C-C composite	∅ 4.5 mm pin with ∅ 6.35 mm spherical heads of 3-D C-C composite	50 g load sliding speed 0.02 m/s in air and 0.04 m/s in vacuum	room temperature and 1000°C	ambient air, vacuum $6,7.10^{-7}$ Pa at room temperature and 67.10^{-7} Pa at 1000°C	deployable solar array	in ambiant air friction coefficient is about 0.28 ; in vacuum at room temperature the coefficient quickly decreases to 0.19 ; in vacuum at 1000°C the coefficient fluctuates from 0,26 to 0,4
			200 g load sliding speed 0.02 m/s in air and 0.04 m/s in vacuum				in ambiant air friction coefficient is about 0.22 ; in vacuum at room temperature the coefficient quickly decreases to 0.12 ; in vacuum at 1000°C the coefficient fluctuates around 0.22
14	c-BN coating thickness 0,4 μm, on a silicon wafer (disc)	Stainless steel ring (SUS 304)	9,8 N for 1h or 10 min 50 rev/min	400°C	ambient air and vacuum $1,7.10^{-3}$ Pa	c-BN coatings are expected to be used as solid lubricants for space applications	in ambiant air friction coefficient is about 0.35; in vacuum at 400°C, the coefficient rapidly reduced to 0.009 at stable stage
			9,8 N for 1h 50 rev/min	600°C	vacuum $1,3.10^{-3}$ Pa		Friction coefficient 0,7 (high because the properties of SUS 304 changed in these conditions) Adherents (SS) on the c-BN coating surface

Table 11f: data from turbine and aerospace industry (following) - [7]

Publication	Matérial		Motion	Temperature	Environment	Application	Results
	Part 1	Part 2					
15	ring 2 mm thickn ∅outside 9 mm inconel 625	cylinder ∅ 8 mm inconel 625	load 14 N, slip amplitude 110µm, frequency 16.6 Hz, number of cycle 4.10^4	20°C to 500°C	$1,3.10^{-3}$ Pa to 10^5 Pa	Nuclear fusion reactor JT-60 of JAERI	The coefficient of friction at 10-3 Pa (1.6-1.8) is independent of temperature an about three times higher than at ambiant air
16	spherical rider single-crystal aluminium oxide (sapphire) ∅ 3,2 and 6,4 mm	amorphous alloy foil	0.2 N 3 mm/min sliding distance 2-3 mm	room temp. to 750°C	vacuum 10 nPa		under 350°C the friction coefficient increase from 1-1.5 to 2-2.5 from 500°C to 750°C the coefficient is very low about 0.3-0.4
17	10 mm thick alacrite disk with silver coating	∅ 8 mm aluminia ball average surface roughness 0.03 µm	1N load sliding velocity 0.02 m/s 5000 cycles	room temperature and 500°C	ambient air	space shuttles HERMES applications	coefficient of friction is lower at 500°C than at room temperature for a thickness less than 2 µm; the reverse situation prevailed when the thickness was greater than 2 µm. The minimum value of 0,15 to 0,20 was obtained for 4,8 µm thick at RT and 1 to 2 µm thick at 500°C.
	10 mm thick alacrite disk with calcium fluoride film		1N load sliding velocity 0.02 m/s 5000 cycles	500°C	ambient air		the average coefficient after 5000 cycles is 0.6-0.7 for the thinnest films and decreased progressively to 0,4 as the film thickness ↗. The critical thickness (f mini) was about 1 µm.
	10 mm thick alacrite disk with chromium carbide / calcium fluoride bilayer (Cr_3C_2/CaF_2)		various loads sliding velocity 0.02 m/s	500°C and 700°C	ambient air		Good results were obtained at 700°C with the Cr_3C_2 3µm/CaF_2 3µm coating : static and dynimic friction coefficient respectively 0.25 and 0.28

Table 11g: data from turbine and aerospace industry (following) - [7]

Publication	Matérial Part 1	Matérial Part 2	Motion	Temperature	Environment	Application	Results
18	Si_3N_4 pin ∅ 12.7 mm	ceramics : SiC, TiC, ZrO_2, Al_2O_3 and Si_3N_4	unlibricated conditions 0.2 m/s normal load 6.14 N Sliding dist. 2000 m except at 600°C : 300 m)	room temperature,200°C, 400°C and 600°C	nitrogen gas atmosphere compared to vacuum		high friction coefficient (about 1) decreasing with the presence of wear particle between surfaces in sliding contact. Wear rate at 600°C is lower in nitrogen gas than in vacuum, except for Si_3N_4 and SiC disks.
19	substrate AISI 304 stainless steel ∅ 25 x 7 mm^2 plates coatings ZrO_2-CaF_3 composite coating deposited by LPPS and YPSZ	∅ 10 mm Al_2O_3 ceramic sphere	30-80 N load 1 mm stroke 1h test duration 10 Hz frequency	room temperature to 800°C	laboratory air	plain journal bearings, space shuttles, hot sections of small jet engines	The ZrO_2–CaF_2 composite exhibited a distinct improvement in wear resistance and frictional characteristics as compared to YPSZ coating at elevated temperatures. At room temperature, the friction coefficient of the ZrO_2–CaF_2 composite decreased with increasing load from 30–80 N. But this situation was reversed for wear loss at room temperature. At 600 or 700°C, the composite exhibited lower friction and wear than at room temperature, 400 and 800°C.
20	PS300 (a metal bonded chrome oxide coating with Ag and BaF_2/CaF_2 lubricant additives)	Al_2O_3 (ceramic) pin 25.4 mm radius of curvature	1m/s sliding velocity 4.91 N load 30 min duration test	25°C 500°C 650°C	air		friction coefficient Al2O3 :about 0.62 at 25°C about 0.32 at 500°C about 0.19 at 650°C
		inconel X-750 (metal) pin 25.4 mm radius of curvature					friction coefficient IncX750 about 0.23 at 25°C about 0.29 at 500°C about 0.31 at 650°C

References

1. Klaffke, D., T. Carstens, and A. Banerji, *Influence of grain refinement on the high temperature fretting behaviour of IN 738 LC.* Wear, 1993. **160**(2): p. 361-366.
2. Miyoshi, K., B.A. Lerch, and S.L. Draper, *Fretting wear of Ti-48Al-2Cr-2Nb.* Tribology International, 2003. **36**(2): p. 145-153.
3. Cox, J.M. and M.G. Gee, *Hot friction testing of ceramics.* Wear, 1997. **203-204**: p. 404-417.
4. Ouyang, J.H., S. Sasaki, and K. Umeda, *The friction and wear characteristics of low-pressure plasma-sprayed ZrO2-BaCrO4 composite coating at elevated temperatures.* Surface and Coatings Technology, 2002. **154**(2-3): p. 131-139.
5. Ouyang, J.H. and S. Sasaki, *Microstructure and tribological characteristics of ZrO2-Y2O3 ceramic coatings deposited by laser-assisted plasma hybrid spraying.* Tribology International, 2002. **35**(4): p. 255-264.
6. Krumpiegl, T., et al., *Amorphous carbon coatings and their tribological behaviour at high temperatures and in high vacuum.* Surface and Coatings Technology, 1999. **120-121**: p. 555-560.
7. Hisakado, T. and H. Tani, *Effects of elevated temperatures and topographies of worn surfaces on friction and wear of ceramics in vacuum.* Wear, 1999. **224**(1): p. 165-172.
8. Yu, Z., K. Inagawa, and Z. Jin, *Tribological properties of TiN and TiC films in vacuum at high temperature.* Thin Solid Films, 1995. **264**(1): p. 52-58.
9. Habig, K.-H., *Fundamentals of the tribological behaviour of diamond, diamond-like carbon and cubic boron nitride coatings.* Surface and Coatings Technology, 1995. **76-77**(2): p. 540-547.
10. Yinglong, W., J. Yuansheng, and W. Shizhu, *The analysis of the friction and wear mechanisms of plasma-sprayed ceramic coatings at 450 °C.* Wear, 1988. **128**(3): p. 265-276.
11. Sliney, H.E., *Solid lubricant materials for high temperatures--a review.* Tribology International, 1982. **15**(5): p. 303-315.
12. XIAO, H. and T. SENDA, *Friction and wear of silicon carbide at elevated temperatures.* Journal of the Ceramic Society of Japan, 1997. **105**(9): p. 750-755.
13. KUSTAS, F.M., et al., *Tribological performance of a 3-D carbon-carbon composite. II: Vacuum environment.* Lubrication engineering, 1996. **52**(2): p. 149-153.
14. Yu, Z., K. Inagawa, and Z. Jin, *Tribological properties of c-BN coatings in vacuum at high temperature.* Surface and Coatings Technology, 1994. **70**(1): p. 147-150.
15. Iwabuchi, A., *Fretting wear of inconel 625 at high temperature and in high vacuum.* Wear, 1985. **106**(1-3): p. 163-175.
16. Miyoshi, K. and D.H. Buckley, *Microstructure and surface chemistry of amorphous alloys important to their friction and wear behavior.* Wear, 1986. **110**(3-4): p. 295-313.
17. ZIMMERMANN, C. and Y. PAULEAU. *Sputter-deposited lubricant thin films operating at elevated temperatures in air*. in *82nd Meeting of the AGARD SMP on "Tribology for Aerospace Systems"*. 1996. Sesimbra, Portugal.

18. HISAKADO, T. and T. KOBAYASHI, *Effects of elevated temperatures on friction and wear mechanisms of ceramics in nitrogen gas.* Japanese journal of tribology, 1999. **44**(6): p. 611-623.

19. Ouyang, J.H., S. Sasaki, and K. Umeda, *Low-pressure plasma-sprayed ZrO2-CaF2 composite coating for high temperature tribological applications.* Surface and Coatings Technology, 2001. **137**(1): p. 21-30.

20. DellaCorte, C., *The effect of counterface on the tribological performance of a high temperature solid lubricant composite from 25 to 650[deg]C.* Surface and Coatings Technology, 1996. **86-87**(2): p. 486-492.

ANNEX 1: Summary from FZJ [6]

Surface Treatments and Coatings for Friction and Wear Resistance

Survey of work performed for the German HTR Nuclear Process Heat Project, original German version by Rodica Exner, formerly Hochtemperaturreaktorbau GmbH

Abbreviated English version by P J Ennis, Research Centre Juelich, IWV2, 52425 Juelich, Germany

Note: the opinions expressed in this summary report are those of the original author

Introduction

A comprehensive survey of the anti-friction and wear resistant surface treatments and coatings work performed for the German HTR Nuclear Process Heat (PNP) Project has been made available. A full translation is in preparation, but in the meantime, a shortened version has been requested as part of the contribution of FZJ to the HTR-E group.

The original document is 269 pages long and includes an extensive literature survey on commercial surface treatments and coatings for a wide variety of materials with 170 references to published work. All the references are listed at the end of this summary, those mentioned in the summary being shown in bold type. In this summary, the experimental results for the surface treatments, coatings and materials that were of interest for the German HTR materials programmes will be outlined. It is intended that the data will provide useful guidance for the development of components for modular HTR designs.

The techniques for modification of the surface properties of materials may be divided into two main types, surface treatments, in which the original dimensions of the component are more or less maintained, and coatings, whereby an external layer is produced on the component.

The main surface treatments and coatings (ST&C) work was on anti-friction and wear resistance of various critical PNP plant components that undergo sliding contact, such as thermal expansion joints and valves, or are subject to fretting as a result of vibrations. Some effort was also put into the assessment of corrosion resistant coatings, the development of thermal barriers, and the potential for systems resistant to hydrogen and tritium permeation. This summary will be restricted to the friction and wear behaviour, as these are of significance for the modular HTR components.

Surface Treatment Techniques

The effectiveness and applications of high temperature coatings are summarized in Table 1 (based on references 2.40, 4.45, 4.46, 4.49-4.52).

type of coating	application/effectiveness
aluminium coating (spray or dip)	corrosion protection of furnace parts, flue pipes – 2-10 times service life extension
enamelling	scaling protection for flue pies and chimneys, boiler tubes
aluminising (Fe-Al coating)	protection against SO_2, SO_3, H_2S for Fe or Ni alloys in heat exchangers
Ni-Cr-Al-Mo-Fe spray coatings	protection against oxidation and chemical attack of boiler components
4-layer spray coatings Ni-Cr / Ni-Cr + ZrO_2-MgO / ZrO_2-MgO / silicon resin	protection of industrial burners against hot gas corrosion 1200 – 1500°C
MCrAlY spray coatings	hot gas corrosion protection in gas turbines, 1100°C
Ni-Al_2O_3 cermet	corrosion protection of blowers in blast furnaces
ZrO_2 or Cr_2O_3 spray coatings	protection against slag and molten metal attack
Al_2O_3 + TiO_2/Al_2O_3-NiAl spray coatings	corrosion protection of casting moulds
sprayed Al_2O_3 or carbides	erosion protection of moulds
boronising, nitriding	corrosion protection of Ti and Ti alloys in Al melts, up to 800°C
sprayed ZrO_2 – Y_2O_3	corrosion protection against V-containing combustion gases
sprayed ZrO_2 – Y_2O_3 + NiCoCrAlY	thermal barrier coating og gas turbine blades
sprayed Mo – Cr_2O_3 + CeO_2 or dispersion coated Ni - SiC	thermal barrier and improved service life in internal combustion engines
Cr-Ni weld cladding or sprayed stellite coatings	exhaust valves in internal combustion engines
aluminising, chromising, or Cr_3C_2 – Ni-Cr coatings	corrosion protection in flue gas systems wear resistance in nuclear plants
oxide scales on components by special thermo-chemical treatments	reduction in permeation of H_2, D_2, T_2

The surface treatment techniques that were considered for PNP components are listed in Table 2.

Table 2
Surface treatments considered for PNP components

	influence on sub-strate			size				dur-ation		repair		application area				
	strength increase	distortion	no distortion expected	large	small	chamber needed	reproducibility (automation)	short (minutes)	long (hours)	no	yes	friction and wear	corrosion	permeation	thermal insulation	not suitable
mechanical hardening	+		+	+			+	+			+		+			
electrochemical etching	+		+	+		+	+	+			+		+			
surface hardening	+	+		+			+		+	+						+
surface glazing			+		+		+	+		+		+				
electron/laser beam hardening			+		+		+	+		+		+				
surface alloying			+		+			+				+				
ion implantation			+		+	+	+	+		+		+		+		
plasma treatment		?	+		+	+	+	+	+			+				
thermochemical	+	+		+		+	+		+			+	+	+		

Surface Treatments for Tribological Properties of PNP Components

Investigations of surface treatments for improvement of the friction and wear characteristics of HTR PNP components were carried out, both in laboratory tests on small specimens and on components under HTR specific conditions.

The parameters that influence the tribological behaviour are:
- test environment (oxidizing, reducing, ...)
- component material
- geometrical form
- surface condition
- pressure
- movement characteristics
- temperature
- time.

Because of the large number of influencing factors, comparison of experimental data becomes very difficult and the behaviour of surface treated materials under service relevant conditions can only be expressed in terms of general tendencies.

The friction and wear behaviour of PNP components may be divided into three temperature regions: below 700°C, 700 – 850°C and above 850°C. The treatments that may be applicable in the three temperature regimes are different and will be discussed separately in the following sections.

Friction and Wear Properties of PNP alloys at Temperatures Below 700°C

The tribological results obtained for various material combinations tested under HTR conditions are summarized in Table 2 (references 1.14, 1.15, 4.54 – 4.56).

Table 2
Tribological properties for PNP materials combinations
at temperatures below 700°C

material combination	temp °C	pressure MPa	atmos	static or dynamic	time h	sliding distance m	friction coeff	result
INCONEL 625/ INCONEL 625	700	20	HHT	S	1000	-	-	no galling
NiAg/ INCONEL 625	700	20	HHT	S	1000	-	-	galled
boiler plate H11/ boiler plate H11	400	0.05	HHT	S	2000	-	-	no galling
IN713LC/ NIMONIC 75	600	10	HHT	D	-	<0.1	0.7	galled
Colmonoy 6/ Colmonoy 6	600	50	HHT	D	-	5.2	0.5-0.6	no galling
IN713LC/ X20CrMoV 12 1	350	10	HHT	D	-	2.5	0.7	no galling
IN713LC/ 17CrMo 5 5	480	10	HHT	D	-	2.5	0.75	grooves
Mo-TZM/ 17CrMo 5 5	480	10	HHT	D	-	1.5	1.2	local galling
INCONEL 617/ INCONEL 617	680	5	HHT	D	-	0.38	>1	galled
Stellite 6/ Stellite 6 wedge	25	ca 2000	He	D	-	150	0.29	local galling
St 37/ St37	20	-	HTR	D	-	0.035	>1	grooves, scratches, material transfer
HASTELLOY X/ HASTELLOY X	20	-	HTR	D	-	0.008	>1	grooves, scratches, material transfer
Nb alloy/ Nb alloy	20	-	HTR	D	-	0.05	>1	grooves, scratches, material transfer

In HTR fretting tests (4.57) typical boiler tube materials were exposed in a helium atmosphere; the vibration frequency was 50 Hz, the amplitude 0.5 mm and the static load 110 N. All the material combinations tested exhibited local wear and material transfer (Table 3).

Table 3
Fretting test results (4.57)
Frequency 50 Hz, amplitude +/- 0.5 mm, static load 110 N

tube	plate	temperature °C	wear depth mm	comments
St 35.8	H II	350	80	
13 CrMo 4 4	13 CrMo 4 4	500	4	
HT 9	HT 9	520	220	
18-8	18-8-2	600	20	
INCOLOY 800	INCOLOY 800	600	20	
HASTELLOY B	HT 9	500	10	
NIMONIC 80A	INCOLOY 800	600	10	

For many unprotected metals, significant wear occurs in helium atmospheres at low temperatures.

Friction and Wear Properties of PNP alloys at 700 - 850°C

At 800°C in dry, impure helium the need for surface protection has been demonstrated (1.17). Alloy 800 and HASTELLOY X were wear tested with a pressure of 0.7 MPa and the coefficient of friction was found to increase from 1.7 to 2.5. Plastic deformation of the mating surfaces was detected.

In Japan, several alloys were tested in helium at 800 and 1000°C (4.58). The results are summarized in Table 4.

Table 4
Wear behaviour of alloys in helium at 800 and 1000°C

alloy	duration h	HASTELLOY X		INCOLOY 800		INCONEL 601		INCONEL 625	
		800°C	/1000°C	800°C	/1000°C	800°C	/1000°C	800°C	/1000°C
HASTELLOY X	100	x	x	x	x	o	x	x	x
	500	x	x	x	x	Δ	x	x	x
	1000		x		x		x		x
INCOLOY 800	100			x	x	x	x	x	x
	500			x	x	x	x	x	x
	1000				x		x		x
INCONEL 601	100					x	x		
	500					x	x		
	1000						x		
INCONEL 625	100							x	x
	500							x	x
	1000								x

x galling
o no galling
Δ slight galling

In 4.57, a diagram (Figure 1) of the wear characteristics of three material combinations, HASTELLOY X/HASTELLOY X, INCOLOY 600/INCOLOY 600 and INCOLOY 800/HASTELLOY X, is given, with experimental data for durations up to 1000 h). The diagram indicates that for a service life of 100 000 h at temperatures below 690°C, the adhesive loads remain below 50 N,

O ··· Hastelloy X mit Hastelloy X
□ ··· Incoloy 600 mit Incoloy 600
◊ ··· Incoloy 800 mit Hastelloy X

unterhalb einer Betriebstemperatur von 690 °C die Haftungskräfte < 50 N bleiben.

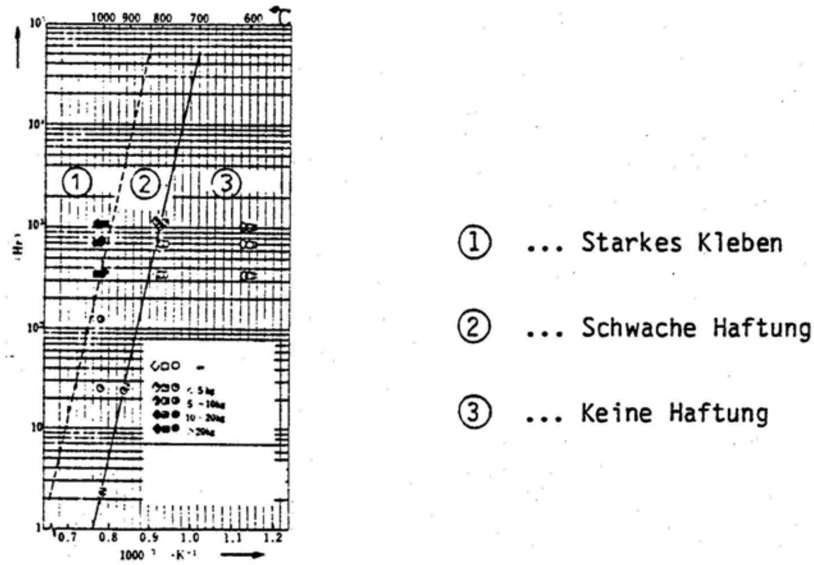


Figure 1
Wear behaviour of alloys in helium:
region 1: galling, region 2: slight galling, region 3: no galling

For the material combination HASTELLOY X/HASTELLOY X in a 20 m friction test, the coefficient of friction μ changed as follows:
μ = 0.88 at 200°C
μ = 0.63 at 500°C
μ = 0.88 at 800°C (*possible typing error in original text!*)

From these values, it was determined that a protective coating is required for service temperatures above 690°C.

Friction and Wear Properties of PNP alloys at > 850°C

For the components under consideration, the following conditions must be fulfilled by a coating system (4.59):

- melting or sublimation or dissociation temperature >1000°C
- phase stability and sufficient strength up to 1000°C
- chemical compatibility with HTR helium, no alloying with substrate
- no diffusion of the individual coating constituents
- sufficient bonding to the substrate, especially under thermal cycling
- no nuclear activation of individual coating constituents

<u>Coatings of pure metals</u>

Table 6 lists the metallic elements that may be considered (melting points <600 K above the maximum service temperature).

Table 6
Melting points of elements for coatings

metal	melting point, °C
Chromium	1800
Hafnium	2207
Iridium	2454
Molybdenum	2610
Niobium	2500
Osmium	2700
Platinum	1773
Rhenium	3147
Rhodium	1966
Ruthenium	2500
Tantalum	2977
Thorium	1827
Titanium	1812
Vanadium	1720
Tungsten	3380
Zirconium	1860

However, the high diffusivity and low strength of pure metals mean that they are not suitable for wear rersistant coatings, as was confirmed by tests on rhodium-coated specimens in air and in helium (4.59, 4.60). The temperature limit for metal coatings lies between 400 and 700°C.

Intermetallics

NiAl-NiCrAlY coatings were extensively tested in the German HTR projects (4.61). However, interdiffusion between coating and substrate led to degradation and coatings frequently spalled off due to the brittleness of the intermetallic compound NiAl.

Hard facing alloys

These materials consist of a Ni-, Co- or Fe-base matrix with a dispersion of very hard particles (boride, carbide, silicide). Tests showed that galling under HTR conditions could be avoided to some extent with alloys of the type Ni-CY-B-SiY and CY-A1 (4.58), but the wear protection of such coatings was not seen as of significance.

Hard materials

Metallic hard materials are the carbides, borides, silicides and nitrides of the transition metals. Non-metallics hard materials are principally oxides.

Cermets

Cermets are composite materials consisting of a hard material in a metallic martrix. Examples are TiN in Ni, Al_2O_3 in Al, CrC in Ni.

Coating Systems Tested Under HTR Conditions

Carbide coatings were investigated by General Atomics in sliding and wear tests (1.12). HASTELLOY X was coated with $Cr_{23}C_6$ or Cr_3C_2. Specimens were tested at 816°C in an HTR helium atmosphere, 3.45 MPa stress, sliding velocity around 8 mm/s. The results were summarized as follows:

1. Coatings with 75-80% chromium carbide ($Cr_{23}C_6$ or Cr_3C_2) provided excellent protection against wear in HTR helium.
2. The coefficient of friction was initially high but decreased to a constant value of 0.4 – 0.5 (see Figure 2)

Tests showed that carbide coatings on Alloy 800 were not as adherent as those on HASTELLOY X.

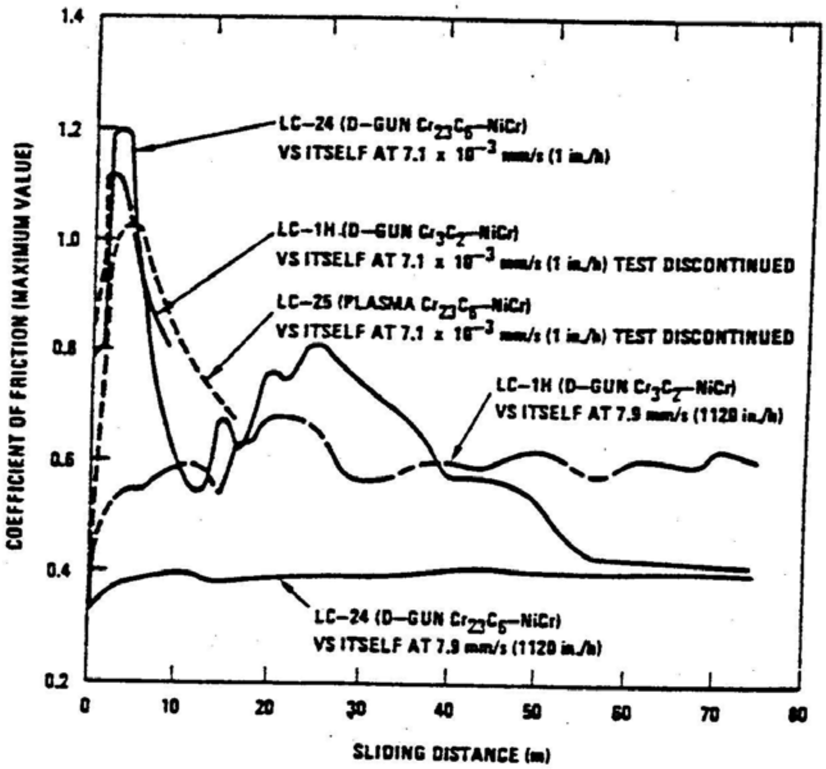


Figure 2
Coefficient of friction as a function of sliding distance (1.12)

From 1976 onwards, HRB carried out testing of coating systems that were considered for HTR applications. Tests comprised wear tests, static adhesive tests, thermal shock and thermal cycling tests, and thermal exposure tests. The carbide coating systems examined are given in Table 7. Coatings were applied by thermal spraying and vapour phase deposition. Iin the following Tables 7 – 13 , the results for various coatings are summarized (4.56, 4.58, 4.59, 4.60, 4.61).

Table 7
Cr_3C_2 coatings

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive /negative	
wear	Cr_3C_2 Duplex	0.98	5	950	5	X	
	Cr_3 C_2 + NiCr	0.5 – 0.8	1 - 5	700	5	X	
	Cr_3 C_2 + NiCr	0.5	20	700	20	X	
static	Cr_3C_2 Duplex	-	10	850	$3 \cdot 10^3$	X	

Experience from CO_2 cooled gas reactors showed that Cr_3C_2 coatings rapidly transform to $Cr_{23}C_6$ and therefore further efforts were devoted to such carbide coatings .

Table 8
$Cr_{23}C_6$ coatings

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive /negative	
wear	Cermet	> 1	5	950	9		X
	Cermet	> 1	3,4	900	0.09		X
	Duplex	0.7	5	950	20	X	
	Cermet	0.83	5	850	20	X	
	Cermet	>1	20	850	1-2		X
	Duplex	0.8	20	850	20	X	
	Cermet	>1	5	700	1.8		X
static	Cermet	-	7	900	2.10^3	X	
	Duplex	-	10	950	3.10^3	X	
	Duplex	-	0.8	1050	100		X
thermal shock	Cermet	-	-		-	X	
	Duplex	-	-		-	X	
ageing	Cermet			950	6.10^3	X	
	Duplex			850	2.10^4	X	
	Duplex			950	$6.5.10^3$		X
	Duplex			950	9.10^3	X	

Table 9
NbC coatings

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive /negative	
wear	NbC-triplex-(NbC-Ni Cr)	spalled	5	950	ca. 5		X
	NbC-duplex-(NbC-NiCr)	0.75	5	950	5	X	
	NbC	spalled	5	950	5		X
	NbC	0.78	20	850	5.3	X	
static	NbC-triplex (NbC-NiCr)	spalled	0.8	1050	100		X
	NbC + Ni	spalled	1	1050	100		X
thermal shock	NbC	-	-		2 cycles		X
ageing	NbC	-	-	950	3.10^3	X	

TiC coatings

Tests carried out in Switzerland (4.6) on TiC coatings in helium at temperatures up to 825°C (Figure 3). Low coefficients of friction were measured at temperatures below 600°C; at temperatures over 600°C the coefficient increased, probably due to softening of the TiC.

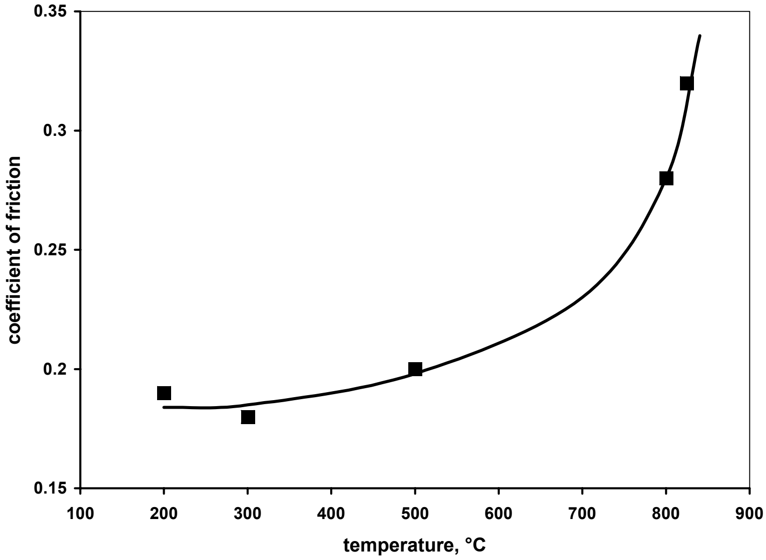


Figure 3
Temperature dependence of the coefficient of friction of TiC in helium (4.6)

Table 10
TiC coatings

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive	/negative
wear	TiC-Triplex-(TiC-TiN-NiTi)	spalled	5	950	20		X
	Ti -Tr ip lex-(TiC-TiN-Ni_3Ti)	spalled.	5	950	5		X
	TiC-Triplex-(TiC-TiN-Ni_3Ti)	0.8-1	5	950	1 3		X
	TiC (Ni_3Ti)	0.3-0.6	5	950	5	X	
	TiC (Ni_3Ti)	0.5	20	850	5	X	
	Ti C + Ti N	>1	5	850	~1		X
static	TiC-Triplex (TiC-TiN Ni Ti)	-	0.8	1050	100	X	
	TiC (Ni_3Ti)	-	0.8	1050	100	X	
	TiC + TiN	-	0.8	1050	100	X	
ageing	TiC Triplex (TiC-TiN Ni Ti)	-	-	950	3. 10^3		X
	TiC (Ni_3Ti)	-	-	950	3. 10^3		X

Table 11
Mo_2C-Mo and W_2C coatings

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive /negative	
wear	Mo_2 C + Mo	1.5	5	850	0.25		X
static	$M0_2$ C + Mo	-	10	1050	100		X
wear	W_2 C	1.2	10	850	0.6		X

Table 12
Nitride coatings

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive /negative	
wear	TiN	>1	5	950	3		X
	TiN	1.5	10	850	0.2		X
	TiN (Ni_3Ti)	>1	5	950	2 19		X
static	TiN (Ni_3Ti)		0.8	1050	100	X	
	TiN (Ni_3Ti)		5	950	$3.\ 10^3$	X	
ageing	TiN			950	$3.\ 10^3$		X (1)
	TiN (Ni_3Ti)			950	$3.\ 10^3$	X	

(1) coating partially oxidized to Ti_2O_3

Table 13
Boride coatings

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive /negative	
wear	Ti B_2 (Mo)	-	0.8	1050	10		X
thermal shock	Ti B_2 (Mo)	-	-		-		X

Table 14
Oxide coatings

type	thermal cycling flat specimen	thermal cycling cylindrical	exposure in He	wear test	ageing 800 °C in He
$Zr0_2$ Plasma	0	0	0	0	0
Al_20_3 Plasma	X		0	X	

O no defects

X cracks and spalling

The influence of a ZrO_2 coating on the coefficient of friction was examined for HASTELLOY X (4.58). The results are shown in Figure 4.

Figure 4
Temperature dependence of coefficient of friction for HASTELLOY X
with ZrO_2 coating exposed in helium (4.58)

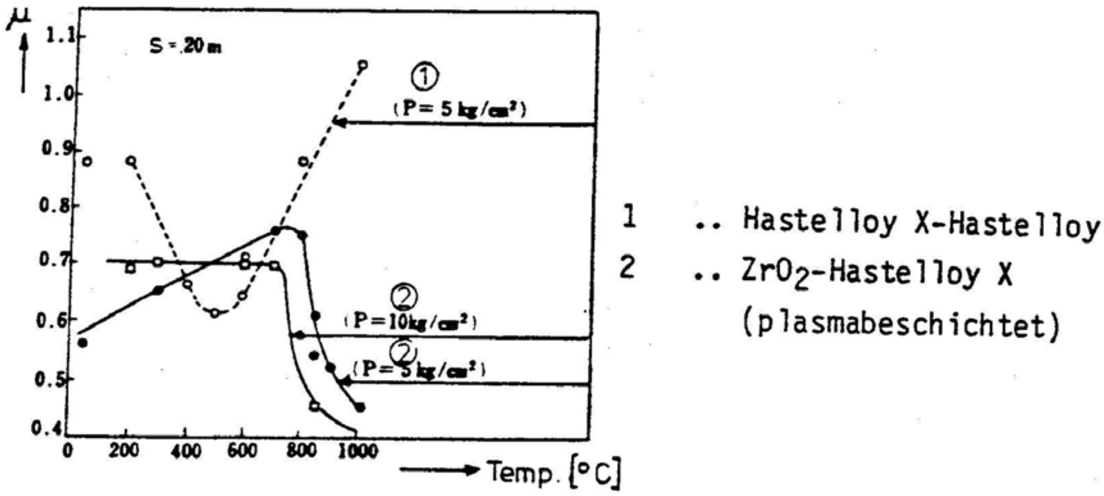


Abb. 4.29 Beziehung zwischen Reibungskoeffizient und Temperatu
in Helium Gas (s = 20 mm) nach /4.58/.

A large number of coatings were investigated by HRB and the results are given in Table 15.

Table 15
Oxide coating systems

test	coating	coefficient of friction	pressure MPa	temp. °C	time, h distance, m	result positive	/negative
static	$M0_20_3$	-	10	850	100		X
wear	Al_20_3	galled	5	950	0.68		X
	Al_20_3 + Ti 0_2	spalled	5	950	-		X
	Al_20_3+ Ti 0_2 (Ni Cr)	galled	5	950	-		X
static	Al_20_3	-	0.8	1050	100	X	
	Al_20_3	-	1	950	$3.\ 10^3$	X	
	Al_20_3	-	10	850	100	X	
	Al_20_3 + Ti02	-	0.8	1050	$3.\ 10^3$	X	
	Al_20_3+ Mg0	-	0.8	1050	100		X
ageing	Al_20_3	-	-	950	$3.\ 10^3$		X
	Al_20_3 - Y_20_3	-	-	950	$3.\ 10^3$	X	
	Al_20_3 - $Zr0_2$	-	-	950	$6.\ 10^3$		X
thermal shock	Al_20_3 (NiCr)	-	-		100 cycles	X	
wear	Cr_20_3 rein	spalled	0.8	1050	100		X
	Cr_20_3 rein	spalled	10	950	100		X
	Cr_20_3 + Al_2O_3	spalled	10	850	5		X
static	Ca0 + $Zr0_2$	spalled	0.8	1050	100		X
wear	MgZ $r0_3$	0.7	5	950	20	X	
	Mg0 + $Zr0_2$	0.58	5	950	5	X	
	Mg0 + $Zr0_2$	0.6	5	950	20	X	
	Mg0 + $Zr0_2$	0.74	20	850	20	x	
static	Mg0 + $Zr0_2$	-	0.8	1050	100	X	
	Mg0 + $Zr0_2$	-	5	950	$10.\ 10^3$		
	Mg0 $Zr0_3$	-		850	$1.\ 10^4$	X	X
thermal shock	Mg $Zr0_3$	-	-		30 cycles	(X)	
wear	$Zr0_2$ cermet	galled	20	850	0.5		X
	($Zr0_2$+Y_20_3 + NiCrAlY) $Zr0_2$ + Ni0	0.9	5	950	20	X	
	$Zr0_2$ + Cr_20_3 (Ni-Cr)	galled	5	950	3.3		X
	$Zr0_2$ + Y_20_3	0.71	5	950	5	X	
	$Zr0_2$ + Y_20_3 (NiCrAlY)	0.8	5	950	20	X	X
	ZrO_2 + Y_20_3 (Ni AlY)	0.54	20	850	20.2	X	
	$Zr0_2$ + Y_20_3 (NiCrAlY + AL_20_3)	galled	20	850	1.4		X
	$Zr0_2$ + Y_20_3 (graded coating NiAlY)	0.58	5	950	20.2	X	
	$Zr0_2$ + Y_20_3 (graded coating Ni AlY)	galled	20	850	20.2		X
	$Zr0_2$ + Y_20_3 (NiCrAlY)	0.5	5	950	100.7	X	
	$Zr0_2$ + Y_20_3 /Yb_20_3	galled	5	950	58		X

	(NiCrAlY)						
	$Zr0_2$ + Ca 0 (Ni Cr)	0.77	5	950	5	X	
	$Zr0_2$ + Ca 0 + Ni0	0.86	5	950	5	X	
	$Zr0_2$ + Ca0 + NiAl	0.88	5	950	20	X	
	$Zr0_2$ + NiAl (Ni Cr)	0.83	5	950	5	X	
	$Zr0_2$ + ZrSi	spalled	5	950	0.3		X
	$ZrSi0_4$	galled	10	950	1.2		X
static	$Zr0_2$ + Cr_20_3 (Ni-Cr) or (Zr0 + Ca0 + NiO)	galled	0.8	1050	100		X
	$Zr0_2$ + Y_20_3		0.8	1050	100	X	
	Z $r0_2$ + Y_20 (NiCrAlY)		0.8	1050	100	X	
	Z $r0_2$ + Y_20 (NiCrAlY)		5	950	$9*10^3$	X	
	$Zr0_2$ + CaO	galled	1	950	3. 10^3		X
	$Zr0_2$ + Ca0 + NiAl	-	1	950	3. 10^3	X	
ageing	$Zr0_2$ Y_20_3						
	(Ni Cr AlY)	-	-	950	6. 10^3	X	
	$Zr0_2$ Y_20_3 (graded coating Ni Cr AlY)	-	-	950	6. 10^3	X	
	$Zr0_2$ Y_20_3 (Ni Cr AlY)	-	-	950	1.2. 10^3	X	
thermal shock	Zr02 (Ni-Cr)	-	-		300 cycles	X	
	Z r02 + Cr 203 (Ni-Cr)	-	-		300 cycles	X	
	Zr02 + Y20 (NiCrAlY)	-	-		300 cycles	X	

The results indicated the following coatings were suitable for applications in the HTR:

- Metallic coatings for temperatures up to 600°C
- Carbide coatings up to 850°C
- Oxide coatings up to 950°C

Table 16 shows the preferred systems and the temperature range of application. Details of the wear resistance of the duplex Zr_2O_3 coatings properties are given in Table 17.

Table 16
Temperature ranges for coatings in HTR helium

coating	coating	Max temp in HTR helium

system	method	200 400 600 800 1000 °C
Colmonoy 6	APS	
Triballoy 700	D-Gun	
Chromium carbide cermet	D-Gun	
Chromium carbide duplex	D-Gun	
Zirkonoxid duplex	APS	
	VPS	

Table 17
Results for duplex Zr_2O_3 coatings (4.66)

coating	test temperature and loading type	time / distance h	m	result
$Zr_2O_3 + Y_2O_3$ (NiCrAlY)	850°C static loading	19	22000	no damage
	950°C static loading	19	25000	no damage
	950°C zero-load exposure	28000	-	no damage
	950°C friction test		100	friction values not increased

Further work

The recommendations regarding further work are listed below:

- Inclusion of chromium carbide in the qualification programme
- Long-term testing of coating systems
- Combined loading effects, such as wear test with superimposed vibration, creep exposed substrates with fretting
- Determination of material loss during wear tests
- Behaviour of specimens coated on one side only
- Influence of component geometry
- Development of quality control : defect tolerance, non-destructive testing
- Determination of adhesion parameters for coatings
- Specification of loading ranges

- Effect of helium impurities

References

(references mentioned in this summary are shown in bold type)

/1.1/ Frost u.a., "Blick durch die Wirtschaft", FAZ 2/78

/1.2/ H. Frey, "Oberflächentechnik", VDI, Mag. 1/85

/1.3/ van Oeteren, "Feuerverzinkung + Beschichtung" Bauverlag GmbH, Wiesbaden Berlin 1983

/1.4/ Battelle - Institut e.V. Frankfurt, 1985

/1.5/ n.n., "Forschungs- und Entwicklungsprogramm Korrosion und Korrosionsschutz", Chem. Ing. Technik 4/85

/1.6/ n.n., "Korrosionsschutz, Fünf Milliarden sparen", Chemische Rundschau 3/86

/1.7/ n.n. "Verschleiß Begriffe, Systemanalyse von Verschleißvorgängen, Gliederung des Verschleißgebietes", DIN 50320

/1.8/ H. Czichos u.a. "Grundvorgänge des Verschleißes metallischer Werkstoffe", VDI-Bericht 194, 1973

/1.9/ T. Arai, "Anwendungsbereiche des TD-Beschichtungsverfahrens in der blechverarbeitenden Industrie, Japans", Bleche, Rohre, Profile 29 (1982) 9.

/1.10/ E. Hintermann "Verschleiß- und Korrosionsschutz durch CVD- und PVD-Überzüge", Metall Heft 3, März 81.

/1.11/ H. Erhart u.a. "Die Oberfläche macht das Produkt", VDI-N. Nr. 19, 5/85.

/1.12/ C.C.Li " Characterization of thermally sprayed coatings for high temperature wear protection applications", Thin Solid Films, 73 (1980) 59-77.

/1.13/ H.W.Fricker, "Fretting in Tube Supports of Heat Exchangers", London, Institution of Mechanical Ingineers, 1972 p. 213-233.

/1.14/ H. Fleischer u.a., "Untersuchung von Funktion und Lebensdauer an Drehdurchführungen, Lagern und Keilmechanismen unter HTR-Bedingungen", Reaktortagung 1978

/1.15/ A.Eiermann u.a. "Untersuchungen von Reibung und Verschleiß in HTR-Helium" Vortrag auf der Reaktortagung 1975.

/1.16/ Steinmüller GmbH und BALCKE-DORR AG, "Auflistung von Reibverschleiß gefährdeten Stellen des RSO Testbündels, des He/He-ZWT's Helix und URKÖ" 8/84.

/1.17/ J.E.Chafey u.a. "Steam generator materials performance in high temperature gas-cooled reaktors", Nuklear technology, Vol 55, Oct. 81.

/2.1/ n.n., "Oberflächentechnik formt den Fugzeugbau", VDI-N 7/85

/2.2/ n.n., Babcock u. Wilcox offers shot-peening process", Nuklear Europa 3

/2.3/ S.Pießlinger=Schweiger, "Elektropolieren hochwertiger funktioneller Edelstahloberflächen", Chemie Technik, Sonderdruck 12 (1983), Heft 4

/2.4/ S.Pießlinger-Schweiger, "Elektropolieren von Edelstahloberflächen für hohe Reinheitsanforderungen", Oberflächentechnik, Surtec 85, Berlin

/2.5/ R.Chatterjee-Fischer u.a., "Oberblick über das Härten mit dem Laserstrahl" HTM 35 (1984)

/2.6/ V.Auerbach u.a., "Randschichtumschmelzen und Randschichtumschmelzlegieren", Oberflächentechnik, Surtec 85, Berlin

/2.7/ G.K.Wolf, "Metallvergüten durch Ionenstrahlen", Chem.-Ing.-Tech 54, 1982

/2.8/ G.K.Wolf, "Verschleiß- und Korrosionsschutz durch Ionenstrahltechniken", Physikalisch-Chemisches Institut der Universität Heidelberg
/2.9/ S.J.Wang, "Industrial application of TiC and TiN coatings for metals by CM and PVD", Oberflächen technik, Surtec 85, Berlin
/2.10/ B.Edenhofer, "Härterei Technische Mitteilung 29, 2/74
/2.11/ Degussa AG, "Salzbadnitrieren nach dem TENIFER-QPQVerfahren", Firmenschrift 1985
/2.12/ Sulzer AG, "Kurzzeit-Gasnitrieren-Neues Wärmebehandlungsverfahren", Werkstoffe und Korrosion 35, 1984
/2.13/ E.Greven, "Werkstoffkunde, Werkstoffprüfung Verlag Handwerk und Technik, B. Ausgabe 79
/2.14/ G.Wahl, "Eigenschaften von Einsatzhärte- und Nitrierschichten", Werkstoffe und Korrosion 35, 1984
/2.15/ W.Domke, "Werkstoffkunde und Werkstoffprüfung" Verlag Giradet, 6. Auflage 75
/2.16/ S.Mareels u.a., "Borieren und Silicieren mit aktivierten Carbiden", Härterei-Tech. Mitt. 40. 1983
/2.17/ H.Kunst, "Beobachten beim Oberflächenborieren von Stahl, III. Borierverfahren. Härterei-Tech. Mitt. 20 (67)
/2.18/ F.Sjoukes, "Die Sherard-Verzinkung", Bleche, Rohre, Profile 26, 1979
/2.19/ n.n., "Möglichkeiten für neue Produkte und Dienstleistungen sowie den Einsatz neuer Technologien in ausgewählten Marktsegmenten" Battelle Institut e.V., Frankfurt, 1985
/2.20/ B.Schweer u.a., "Ionnitrieren-Anlagen und Anwendungsgebiete", Elektrowärme international 34, 12/76
/2.21/ R.Jonck, "Entwicklungstendenzen in der Wärmebehandlungstechnik", Surtec 85, Berlin
/2.22/ n.n., "Nitrierte Zahnräder trotzen dem Verschleiß", VDI-N, 5/86
/2.23/ n.n., "Beschichtungen für höchste Ansprüche", Firmenschrift der Buser AG, 1985
/2.24/ n.n., "Oberflächenschutz" Firmenschrift der Huber und Suhner AG, 1985
/2.25/ E.Schmeling, "Schutz gegen Fressen", KEM 2/86
/2.26/ n.n., "Nickelschicht wird schön und dauerhaft zugleich", VDI-N, 4/85
/2.27/ J.Ehrhardt, "Herstellung und Eigenschaften galvanisch abgeschiedener Dispersionsschichten", Oberflächen-Surface 24, Heft 10, 83
/2.28/ D.R.Gabe, "To electrodeposit or not to electrodeposit", Surtec 85, Berlin
/2.29/ G.Nagel, "Veränderung der Eigenschaften des Grundmaterials durch Aufbringen von galvanischen Schichten", Galvanotechnik Saulgau, 71 Nr. 2, 1980
/2.30/ A.Knauscher, "Schmelztauchverfahren. Oberflächen veredeln und Plattieren von Metallen. VEB, Deutscher Verlag für Grundstoffindustrie, Leipzig 1978
2.31/ T.Arai, "Properties of carbide coated materials by salt bath immersing method, Surtec 85, Berlin
/2.32/ T.Arai, "Anwendungsbereiche des TD-Beschichtungsverf ahrens in der blechverarbeitenden Industrie Japans", Bleche, Rohre, Profile 29, 1982
/2.33/ A.Metthews, "Titanium Nitride PVD Coating Technology", Surface Engineering 1, 1983
/2.34/ H.M.Gabriel u.a., "Herstellung Verschleiß- und Korrosionsmindernder sowie dekorativer Schichten mittels Sputter-ION-Plating", Surtec 85, Berlin
/2.35/ n.n., "Beschichtete Schnellstrahl-Spiralbohrer", Schweizer Maschinenmarkt, Goldach Nr. 20, 1981
/2.36/ W.M.Mularie, u.a., "Cathodic arc deposition- the bridge between atomic and particulate deposition technology", DVS 98

/2.37/ O.Pacher, "Harte Schichten als Verschleißschutz", Materialwissenschaften-Werkstoffkunde, Informationsveranstaltung des Bundesministerium für Wirtschaft und Forschung, Innsbruck 9/82
/2.38/ W.D.Münz u.a., "Thin solid Films, 96, 1982
/2.39/ E.Ertürk, "Thermisches Spritzen" Vortrag BernexVerschleißschutzseminar, 9/83
/2.40/ K.Smolka, "Thermisches Spritzen - Ein Leitfaden für den Praktiker-" DVS Bund 15, 1985
/2.41/ H.R.Eschnauer, "Spritzwerkstoffe und ihre Anwendungen", Vortrag Bernex-Verschleißschutzseminar 9/83
/2.42/ E.Schwarz, "Verschleißfestes Hartbeschichten durch Plasma und Flammschockspritzen (Detonationsspritzen), Werkstatt und Betrieb 110, 1977
/2.43/ N.Wagner u.a., "Herstellung von Verschleißfesten WC-Co-Schichten durch Hypersonic-Flammspritzen", Z. Werkstofftech. 16, 1985
/2.44/ H.Kreye u.a., "Untersuchungen zum thermischen Spritzen von WC-Co mit dem Hypersonic-Spray-Verf ahren", Metall 3 Jahrgang 3, 3/83
/2.45/ H.D.Steffens, "Future aspects of thermal spraying" ITSC' 1983, DVS-Bericht Nr. 80
/2.46/ n.n., "Plasmaspritztechnik-Grundlagen und Anwendungen", Firmenschrift der Plasma Technik AG, Schweiz 1983
/2.47/ R.Bonetti, "CVD-Verfahren" Vortrag Bernex-Verschleißschutzseminar 9/83
/2.48/ W.Schintlmeister u.a., "CVD-Hartstoffbeschichtete Werkzeuge und Verschleißteile", Surtec 85, Berlin
/2.49/ K.M.Eisele. "Die Beschichtung und Strukturierung von elektronischen Bauteilen durch Plasmaverfahren" Surtec 85, Berlin
/2.50/ D.Bäuerle, "Materialbearbeitung mit Laserlicht", Laser und Optolektronik Nr. 1, 1985
/2.51/ W.Hegner, "Unter Pulver-Auftragschweißen mit Bandelektrode", Wahlarbeit RWTH Aachen, 1975
/2.52/ R.Streib, "Persönliche Mitteilungen" GVA Bocholt 10/85
/2.53/ n.n., "Sprengplattierte Bleche von VDM", Firmenschrift VDM AG, 10/85
/2.54/ n.n., "Pfandler mit neuem Unternehmungskonzept", Europa Chemie 5/85
/2.55/ n.n., "Glasemaillierte und Korrosionsfeste Stahltanks auch für den Abwassersektor", Chemie Technik
/2.56/ W.Beier, "Glas aus der Flasche - Sol-Gel-Methode, VDI-N Dez. 1985
/2.57/ n.n., "Neue Werkstoffe" VDI-Magazin 11/8.
/2.58/ W.Hansen, "Oberflächentechnologien für Triebwerkskomponenten - Stand und Tendenzen", Vortrag Surtec 85, Berlin
/2.59/ U.Mürrle u.a., "Eigenschaften und Anwendungen auftraggelöteter Schichten", Schweißen und Schneiden 38 , 1986
/2.60/ H.P.Schreiber u.a., "Corrosion Protection by Plasma-Polymerized Coatings", Thin solid film 72 (1980)
/3.1/ K.H.Kloos u.a., "übertragbarkeit von Probestab-Schwingfestigkeitseigenschaften auf Bauteilen", VDI-Berichte Nr. 354, 1979
/3.2/ H.K.Kloos, "über die Bedeutung der Oberflächenund Randschichteigenschaften für die Bauteilhaltbarkeit", VDI-Berichte 506, 1984
/3.3/ H.D.Steffens, "Dauerschwingfestigkeitsprüfung an Bauteilen mit thermisch gespritzten Überzügen" Vortrag Surtec 85, Berlin
/3.4/ n.n., "Aufdampf- und Zerstäubungsmaterialien Verdampferquellen, Firmenschrift Fa. Balzers AG, Ausgabe 84/86

/4.1/ M. Kübert u.a., "Verschleißschutzschichten für Werkzeuge zum Kaltumformen", Werkstatt und Betrieb 116, 1983
/4.2/ R.Woska, "Oberflächenbehandlung von Werkzeugen der Blechverarbeitung zum Verschleißschutz",
/4.3/ V.Rocek, "Fortschritte bei Werkzeugen" Technische Rundschau Nr. 16, April 1983
/4.4/ V.Denarne u.a., "Verwendung von Hartstoff- und Hartmetallschichten für Schneidwerkzeuge", Vakuum Technik, 32. Jahrgang, Heft 7
/4.5/ n.n., "Beschichtete Schnellstrahl-Spiralbohrer, Schweizer Maschinenmarkt Nr. 20/81
/4.6/ H.E.Hintermann, "Verschleißschutzschichten", Schmiertechnik und Tribologie 5/81
/4.7/ H. Mc Kee, "Coated carbides for the common job", Tooling and Production, April 83
/4.8/ U.König, "Glimmbehandlung von CVD-beschichteten Wendeschneidplatten"
/4.9/ R.L.Hatschek, "Coatings: revolution in HSS tools", American maschinist, 3/83
/4.10/ Fa.Balzers, "Applikationsberichte Hartstoffbeschichtete Werkzeuge", Mitteilungen der Balzers AG
/4.11/ Fa.Sulzer, "Wolframcarbid Verschleißschutz abgeschieden bei 300 - 550 oC", Mitteilungen der Gebr. Sulzer AG, 1981
/4.12/ R.Poreit u.a., "The influence of different CVD coatings carbide coated tool materials", Vortrag Surtec 85, Berlin
/4.13/ E.Moll u.a., "Verschleißschutz mittels ionenplattierter Hartstoffschichten am Beispiel von Zerspannungswerkzeugen", Mitt. aus dem Bereich Dünnschichtmetallurgie des Balzers AG
/4.14/ M.Gierzynsk a-Dolna, "Effect of the surface layer increasing the life of tools for plastic working", Journal of Mechanical Working Technology 6, 82
/4.15/ D.Schmoeckel, "Verschleißverhalten CVD-beschichteter Schneidwerkzeuge", Industrie Anzeiger 11/83
/4.16/ H.G.Joost, "Untersuchungen über die Anwendbarkeit von beschichteten und Oberflächenbehandelten Gesenkschneidewerkzeugen", Dissertation Universität Hannover 1980
/4.17/ T.Auasawa u. a., "Coated high-Speed steel tools for improved thread cutting productivity", Proc. of the 21. internat. machine toole design and res. conf., Vol II -cutting and tribology Univ. of Wales, Swansea, 80
/4.18/ D.Deller, "Laboratory a. field test results an TiN coated high speed steel cutting tools", Presented, IMTS - 82 Technical conference, Sept. 1982
/4.19/ H.Erhart u.a., "Die Oberfläche macht das Produkt", VDI-N 5/85
/4.20/ H.C.Child, "Surface Treatments for Tribology Problems", Seminar held at the Joint Research Centre, CEC Petten Establishmemt, 83
/4.21/ J.Kiefer u.a., "Standzeitverbesserungen durch Oberflächenbeschichten von Werkzeugen", Veröffentlichung VEW, Forschung und Qualitätswesen, Werk Kappenberg, österreich
/4.22/ R.Buhl u.a., "TiN coatings an steel", Thin solid films 80, 1981
/4.23/ K.Häuser, "Werkzeuge zwingen Werkzeugmaschinen zu Höchstleistungen, VDI-N, 10/85
/4.24/ n.n., "Materialwissenschaften-Werkstoffkunde", Bundesministerium für Wissenschaft und Forschung, Innsbruck 9/82
/4.25/ E.Bergmann, "Ionenplattierte Verschleiß-SchutzSchichten", Surtec 85, Berlin
/4.26/ Fa.Gore, "Korrosionsschutz im Apparate- und Rohrleitungsbau", Chemie-Technik 2/85
/4.27/ H.Behnisch, "Mit Schweißplattierungen und Spritzschichten ...", KEM, Dez. 82

/4.28/ E.Tenkchoff u.a., "Verbesserungen und Innovtionen durch neuartige Anforderungen", Atomwirtschaft 3/86

/4.29/ n.n., "Technische Informationen", LWK-Oberflächentechnik GmbH, 1985

/4.30/ Mathesius, "Persönliche Mitteilung, Fa. Bernex, Langenfeld, 8/85

/4.31/ H. Hauff, "Die Oberfläche macht oft erst den Wert eines Teils aus", VDIN-M 1/85

/4.32/ H. Kemper, "Persönliche Mitteilung", Fa. Bernex, 8/ 86

/4.33/ B.Grundman, "Persönliche Mitteilung, Fa. CastolinEutectic, 9/85

/4.34/ P.C.Wolf, "Neue Anwendungsgebiete des Plasmaspritzens in der Energietechnik", Wärme Band 89, Heft 3, 1981

/4.35/ G.Nagel, "Rohstoffe sparen- Flugzeugteile galvanisch beschichten", Industrie Anzeiger Nr. 59, 7/82

/4.36/ G.Nagel, " Veränderungen der Eigenschaften des Grundmaterials durch Aufbringen von galvanischen Schichten", Galvanotechnik 71, 1980

/4.37/ n.n., "Oberflächentechnik formt den Flugzeugbau", VDI-N 7/85

/4.38/ H.W.Grünling u.a., "Bedeutung von Oxidschichten für die moderne Hochtemperaturtechnik", Deutsche Gesellschaft für Metallkunde e.V., Oberursel, 1983

/4.39/ H.W.Grünling, "Schutzschichten für Hochtemperaturanwendungen", 3. Arbeitstagung 1984, angewandte Oberflächenanalytik

/4.40/ R.Rimanek, "Auskleidungen und Beschichtungen von Apparaten mit Kunststoffen", 3 R international Heft 4, April 1982

/4.41/ C.Iten, "Die Beeinflussung der Eigenschaften von funktionellen Nickelschichten", Der Machinenschaden 57, 1984

/4.42/ n.n., "Technische Informationen, LWK Oberflächentechnik, Gummersbach 1985

/4.43/ P.C.Wolf, "Neue Anwendungsgebiete des Plasmaspritzens in der Energietechnik", Wärme Band 89, Heft 3, 1981

/4.44/ n.n., "Die Dickvernickelung nach dem Toraxier-Verf ahren, Firmenschrift von Roll 1984

/4.45/ n.n., "Sch ichtkennwerte", Schrift der Fa. Union Carbide, 1983

/4.46/ H.Eschnauer, "Fortschritt bei thermischen Spritzen" Metall 39, Heft 3, 85

/4.47/ A.Matthews, "Alumina and titanium carbide coatings deposited by ionisation assisted reactive physica

/4.48/ n.n., "Fülldraht", Katalog der Corodur Fülldraht GmbH, 1985

/4.49/ A.Appen u.a., "Hitzebeständige Korrosions-, Wärme- und Verschleißschutzschichten", VEB Deutscher Verlag für Grundstoffindustrie, Leipzig, 1984

/4.50/ J.Peters u.a., "Bildung von Aluminiumoxid-Deckschichten aus Eisenbasislegierungen", Werkstoffe und Korrosion 35, 1984

/4.51/ S.Stecura u.a., "Effects of yttrium, aluminium and chromium concentrations in bond coatings an the performance of zirconia-yttria thermal barriers", NASA Technical Memorandum 79206, 7/79

/4.52/ Offenlegungsschrift DE 3438439 A 1 Pulveroberflächenschweißverfahren" 5/85

/4.53/ H.Möllenhoff u.a., "Oxidschichtbildung beim Prozeß der Kohlevergasung mit HTR-Wärme und ihre Bedeutung für die Wasserstoff- und Tritiumpermeation", Deutsche Gesellschaft für Metallkunde e.V., Oberursel 1983

/4.54/ HRB Bericht NJ 0028 B1. 28 "Ergebnisse der statischen Verschleißversuche in HHT-Atmosphäre (78)"

/4.55/ HRB Bericht NJ-GHRA 000 801, Seite 16, "Dynamische Reibversuche in HTR-Atmosphäre"

/4.56/ R.Engel u.a., "Ädhäsiver Verschleiß metallischer Werkstoffe und das Verhalten von Schutzschichten in HTR-Helium" HRB Jülich/Mannheim, Fachinformation Energie, Physik, Mathematik 1982

/4.57/ H.W.Fricker, "Schwingungsverschleiß in Rohrhalterungen von Wärmetaucherrohren", Sonderheft, Nuclex 1972

/4.58/ Koichi Murase u.a., "Research and Development of HTR related technology, ISSN 0014-5646, No. 99, Dec. 81

/4.59/ Demus u.a., "Reib- und Verschleißschichten im PNP Projekt, HRB Bericht PNP-5170-Bf-GHRA 001076, Juli 84

/4.60/ W.Thiele u. a., "Entwicklung von Schutzschichten zur Verhinderung von primärseitiger Korrosion, Reibung und Verschleiß", Schriftreihe "Energiepolitik in Nordrhein-Westfalen" Bd. 14, 3/82

/4.61/ HRB, Tabellenwerte aus PNP und HHT Jahresberichten bis 1984

/4.62/ A.R.Nicoll u.a., "The behaviors of air plasma sprayed zirconia in high temperature reactor helium", Thin Solid Films, 95, 1982

/4.63/ H.Römpp, "Chemie Lexikon", Frank'sche Verlagshandlung Stuttgart, Fünfte Auflage 1962

/4.64/ Theymann "Schutzschichten gegen Reibung und Verschleiß" HRB Bericht PNP-5170-BF-GHRA-000839

/4.65/ W.W. Sytschew u.a., "Temperatureinfluß auf Reibung und Verschleiß der auf Molybdän aufgetragenen Boridüberzügen" UDK 546.271 : 539.23, 1974

/4.66/ Mitteilungen auf der Sitzung UG 1 des Arbeitskreises 8 der EG-HTR in Jülich 6/86

/5.1/ R. Blevins, "Fretting wear of heat exchanger tubes" G.A. Report GA-14817, 4/78

/5.2/ PNP 500 Planungsblatt, IA-Nr. 4800002.5 D von 5/83

/5.3/ PNP-Modul mit WKV, IA-Nr. 78.10612.0 von 5/85

/5.4/ BALCKE-DORR AG, Berechnungen URKÖ 10 MW, Blatt 1 und 2, 1984

/5.5/ BALCKE-DÜRR AG, Ergebnisse der FE-Berechnungen, URKÖ 125 MW, 1984

/5.6/ BALCKE-DORR AG, Ergebnisse der FE-Berechnungen, URKO 170 MW, 1985

/5.7/ H. Öry, "Abschlußbericht über die Beurteilung des Schwingungsverhaltens der Rohre des URKÖ-Versuchsbündels", 11/83

/5.8/ Steinmüller GmbH, "Verschleiß-gefährdete FLächen des PNP He/He-ZWT's (Helix); Steinmüller Aug. 84

/5.9/ BALCKE-DüRR AG, "PNP He/He-ZWT, Fretting gefährdete Stellen des URKO-WT), BALCKE-DÜRR AG 7/84

/5.10/ Zipper, "Aufgabenspezifkation zur Untersuchung von beschichteten Bauteilen einer PNP-Anlage" Interatom-Notiz vom 06.10.1983

/5.11/ Europäische Patentanmeldung, "Rohrhalterung in einer öffnung einer Platte; V-Nr. 0185902 vom 02.07.1986

/5.12/ n.n., "Planung und Entwicklung einer Nachwärmeabfuhrkühlers (NWA)", Jahresbericht 1985 der Fa. BALCKE-DÜRR AG

/5.13/ R. Exner u.a., "Schichten gegen Reibung und Verschleiß bei einem Hochtemperatur-Helium/Helium-Zwischenwärmeaustauscher für PNP-Anlagen", Chem.-Ing.Tech. 57, 1985

/6.1/ H. Demus, "Technische Spezifikation-Luftplasmagespritztes Schichtsystem. Zr02 (Y203) NiCrAlY -PNP 5179-CB-GHRA 000177 A, 5/ 83

/7.1/ H. Bodmann u.a., Ad hoc-AK-Reib- und VerschleißSchutzschichten 1. Sitzung PNP-5177-BB-GHRA 000965, 6/84

/7.2/ Demus u.a., "Reib- und Verschleißschutzschichten im PNP-Projekt, PNP-5170 BF-GHRA 01076, 7/84

/7.3/ Böhlo u.a., "Bau teilbezogene Reibversuche", PNP-5177 BE-GHRA 00486, 8/83

H. Bodmann u.a., "Reib-/Verschleiß-Schutzschichten, Stand", Bestandsaufnahme und Bewertung der von der HRB durchgeführten Entwicklungen, PNP-5170-BA-GHRA 00190, 10/84

/7.5/ E. Schwarz, "Einsatz von UCAR-Cermet und Duplex-Schichten in Heliumsystemen", Mitteilung der Fa. Union Carbide 1/83

/7.6/ Mittenbühler, "Beschichtungen in der Großanlage KVK und deren Zustand nach erstem Einsatz bei 950 °C, Notiz vom 30.07.1984

/7.7/ D. Steffens u.a., "Methods based an ultrasound and optics for the nondestructive inspection of thermally sprayed coatings", Thin solid Films, 83, 1981

/7.8/ BALCKE-DÜRR AG, Tabellen-Reib- und Verschleißgefährdete Flächen, 7/84

/7.9/ H. Schuster u. a., "Gas-Metall-Reaktionen im Primärgas des Hochtemperaturreaktors", Aufbau von Oxidschichten auf Hochtemperaturwerkstoffen und ihre technische Bedeutung, Deutsche Gesellschaft für Metallkunde e.V., Oberursel 1983

/8.1/ G. Weirich u. a., "Wirtschaftliches Panzern durch Spritzen und Schmelzverbinden von Metallpulvern mit der Flamme", Technica Heft 23, 1983

/8.2/ H. Benninghoff, "Breite Palette prägt Oberflächentechnik", VDI-N 2/85

/8.3/ R. Kötz, "Voller Möglichkeiten für morgen", KEM 6/85

/8.4/ n.n., " Chemische Industrie und Plastikverarbeitung", Schrift der Fa. Union Carbide 83

/8.5/ n.n., "Wirtschaftliches Veredeln von Formteilen", Informationsschrift der Fa. Leybold-Heraeus GmbH

ANNEX 2: Recommendation for coatings in the frame of WP4 / D33

(Doc. ref NFTW – IBV / 04.010)

RECOMMENDATIONS for COATINGS to be provided FOR BRUSH SEALS

Deliverable 33 for WP4 work of HTR-E: "Feasibility study for a helium rotating shaft seal for turbogenerator".

RECOMMENDATIONS for COATINGS to be provided FOR BRUSH SEALS
Doc. ref NFTW – IBV / 04.010

This paper was required in the frame of the Deliverable 33 for WP4 work of HTR-E: "Feasibility study for a helium rotating shaft seal for turbogenerator".

1. Introduction

The feasibility problem was how to ensure the tightness of a rotating seal described by a 450mm diameter shaft immersed in helium at 110°C and 26 bar pressure, located at the turbine end, and rotating at a 3000rpm speed. The study suggested the combination of a labyrinth seal and a brush seal. The seal consisted in thousands wire bristles with 0.1mm approx. diameter. The bristles can be made of low alloy steel, stainless steel, or cobalt or nickel-based superalloy. This solution brings several advantages, but these brushes made of metallic bristles are rubbing onto the rotor. Rubbing will lead to wear, but the wear of the rotor has to be avoided whereas worn brushes can be changed. This paper aims at listing the coatings which can be used to protect the rotor surface.

2. Rotor and brushes operating conditions

The normal and exceptional operating conditions would be the following one:

- operating fluid : helium with active impurities,
- operating pressure : 26 bar,
- operating temperature (near the leak) : 110°C,
- normal design temperature : 150°C,
- exceptional transient temperature (on a short duration) : 450 to 500°C,
- operating rotation speed : 3000 tr/mn (linear speed ≈ 80 m/s),
- shaft diameter : 450 mm minimum,
- axial clearance: 0.5 + ≈ 10 = 10.5 mm,
- radial clearance : 0.4 mm,
- seal lifetime : 60 years with inspection and regular replacement of parts (as infrequently as possible)

3. Possible materials for rotor shaft and brushes

Rotor shaft
It is usually made in high alloyed steel, and presents a high ultimate tensile strength, a good creep resistance, a good resistance to fatigue and wear.

Presently, no material specification is available, but the material will probably present good structural properties and not necessary, good surface resistance. It is then envisaged to give it a higher surface resistance with a hard coating which will protect the surface against wear.

Brushes:
The bristles of the brush can be made of low alloy steel, stainless steel, or cobalt or nickel-based super-alloy. The objective is to have the bristles worn, instead of the rotor shaft, since brushes can be more easily changed. The cobalt alloy presents a low nuclear compatibility. If brushes have to be changed quite frequently, the cost impact of nickel-based super-alloy brushes has to be considered.

To conclude on brushes, a first approach could consider only steel alloy and Stainless Steel as bristle material.

4. Possible materials and coatings for the rotor shaft in helium environment

The review of materials and coatings which was the subject of deliverable D39 for WP5 [1], showed that few materials can be used in rubbing conditions, without a coating even at low temperatures (110-150°C). The temperature range which was considered in this paper, is 20-500°C, due to 110-150°C for normal operating conditions and temperatures up to 500°C for exceptional conditions.

4.1. Materials without coating

The D39 review showed that few materials can be used in rubbing conditions, without a coating even at low temperatures, in Helium environment.
In PNP environment, the only data available at temperatures below 700°C giving combinations which were working, were the following:

Material combination	Temperat. (°C)	Helium pressure (MPa)	Sliding distance (m)	Friction coeff.	Result
Colmonoy 6 / Colmonoy 6	600°C	50	5.2	0.5-0.6	No galling
Inc 713LC / X20CrMoV12-1	350°C	10	2.5	0.7	No galling
Hastelloy X / Hastelloy X	200°C	0.7	20	0.88	No galling
Triballoy 700 / Triballoy	600°C	50	-	-	No galling

* Triballoy :base Ni, + Mo, Cr, Si with 50 % constituted of lave phase in a hard matrix

It has to be noted that these tests were realised on a short distance (equivalent to less than 2 rotor turn, 4 and 14 turns – shaft diameter = 0.45m). Nevertheless, whereas other materials showed grooves and scratches in the first meters, the material combinations given in this table showed no galling but high friction coefficient. They are not structural materials for rotor shaft, but some of them (colmonoy 6, hastelloy, triballoy 700) can be used as a coating.

Other materials such as the following, were galling and showed grooves or scratches in dynamic conditions and HTR helium environment. For instance, Stellite 6 which is a frequently used alloy, presented bad results at low temperatures in helium environment.

Material combination	Temperature (°C)
Inc713LC / 17CrMo5-5	480°C
Mo-TZM / 17CrMo 5-5	480°C
Stellite 6 / Stellite 6	25°C
Stellite 37 / Stellite 37	20°C

Remark: in Sodium (Na) environment the results for these same materials were not very satisfactory either.

For major of tested unprotected metals, significant wear occurred in Helium atmosphere at low temperatures. The review confirmed that few materials have a good behaviour without a coating, and that others, including **structural material, need a coating to keep their metallurgical properties and ensure surface properties. Few coatings are reviewed hereafter.**

4.2. Coatings between 20-500°C

The review showed several examples of coatings able to fulfil our requirements. The following coatings were reported as presenting a good behaviour in a 20-600°C temperature range.

- Metallic coatings such as Triballoy 700, Hastelloy or Colmonoy 6

- Triballoy 700: it can be deposited from powder under a thick deposit: 1-2mm, or thin deposit by D-Gun (thickness < 0.1mm), but the process is property of few companies (from NNC)
- Colmonoy 6: deposited by PTA, TIG, … from powder (thick deposit: 1-2mm)
- Hastelloy can be deposited from solid electrode (thick deposit: 1-2mm)

In Sodium environment:
Results from FBR bibliography and Na environment showed that coating properties and resistance widely varied from one manufacturer to an other (ex in D37 between Leguellec and SNM). A bad coating can result in bad results in the same range of solicitations and temperatures. Best results probably due to a higher density of the deposit, gave a friction coefficient included between 0.3 and 0.55 and low wear in Na environment.

In Helium environment:
To compare in helium, friction coefficients were given in the range 0.5 - 0.88 (see same materials in the previous paragraph).
It ought to behave as hard coatings, with wear rather located on brushes. Nevertheless, it has to be confirmed by tests.

- TiC coating : low friction coefficients (<0.25) in pure Helium (T<600°C).

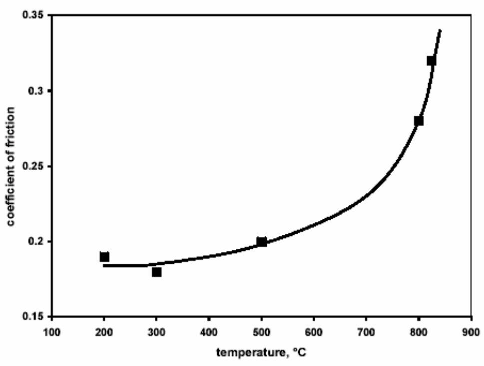


Figure 3
Temperature dependence of the coefficient of friction of TiC in helium (4.6)

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This result was also found in Na environment (FBR) at 180 and 560°C, and in vacuum environment at 400°C.
The TiC can be deposited by PTA or Vapor deposition technique. It's a thin coating (thickness < 0.1mm),

- <u>Tungsten carbide</u>

  - A Tungsten carbide coupled with cobalt was reported to be used in gas turbine components, to protect against sliding labyrinth seal fins and different components of compressors at temperature less than 538°C.
  - From Dragon experience, it was also reported that Titanium or Tungsten carbide were only superficially scored by graphite particles after very long runs. Of the two, Tungsten carbide seemed to have the best behaviour till 550-600°C.
  - Tungsten carbide cermet (WC 25%, WC_3/Cr_2C_3 70%, 5%Ni – D-Gun) – named LW-5. It was quite good in CO_2 environment (AGR) for temperatures less than 400°C. In Helium environment, rubbing configuration LW-5 / LW-5 wore severely at 20°C, but the behaviour improved with temperature increase. In any case, the situation of LW-5 rubbing against bristles has to be verified by tests.

<u>Remark on chromium carbides</u>
Chromium carbide cermet (Cr_2C_3 (80%)/ NiCr (20%) D-Gun)

This chromium carbide cermet (LC-1B) was largely used in CO_2 environment (AGR), with quite good results under 200°C.
Other results from Dragon experience showed <u>bad results in Helium for this chromium carbide cermet at lower temperatures</u>.

- Configurations LC-1B or LC-2 like-on-like wore severely at 20°C in pure Helium (0.1 or 5MPa gas pressure) and in impure Helium environment (2MPa gas pressure). Whatever the gas composition, results are better for upper temperatures and wear rate fell with increasing temperatures. The test was conducted between 20 and 800°C. Results at intermediary temperatures were not given.
- LC-1B rubbing on type 316 Stainless Steel gave unsatisfactory results with galling badly, whatever the temperatures in pure helium atmosphere.

Nevertheless, according to NNC, chromium carbide should not be ruled out for this rotor application. It was also recommended to use a Ni binder and not a NiCr binder together with the Cr_2C_3 cermet (FANP – P. Combrade)

- SiC carbide

This SiC carbide is suggested in ref [2] (corresponding to ref 12 of table 11e in D39). At room temperature, it presents a friction coefficient included in the range 0.15-0.7 depending on the applied contact pressure, respectively 0.4 to 1.2 MPa. In any case the friction coefficient is 0.65 at respectively 400 and 200°C.

- Zirconia, alumina coatings

These coatings type $ZrO_2 - Y_2O_3$ or Al_2O_3 are usually used at high temperatures, but some papers (ref 3, 4 and 5 of table 11b in D39 — see ref [3, 4, 5] of this document) indicated this coating for applications in air at low temperatures in the range 20 – 800/1200°C. They showed principally a decrease of the friction coefficient with a decrease of the temperature (ex μ=0.2-0.3 at 200°C increases up to 0.45-0.63 at 400°C). ZrO_2 can be also stabilised with CaO, NiO species, Al_2O_3 with MgO species but not systematically. Al_2O_3 can be used alone.

- Lubricants such as MoS_2 and WS_2

The lubricating effect of MoS_2 and WS_2 is very known in argon or Helium environment, leading to very low friction coefficient on S.S : μ=0.1 at T<400°C and μ=0.22 from 400 to 700°C for MoS_2. Close values for WS_2.
MoS_2 plugs can be installed to maintain a good lubrication, but they can break and be covered with dust.
A more efficient way is to integrate MoS_2 to one of the antagonistic material, as it was done in Dragon project by mixing MoS_2 in the graphite stator rubbing against the Tungsten carbide rotor. The results were really satisfactory on 250h duration test: good for the rotor, excellent for the stator. This result could be adapted to our case, assuming that in this configuration, the graphite ring will probably much wear, but could be changed. Another point to verify is the nuclear compatibility of MoS_2 and WS_2, although it was already used in Dragon reactor.

5. Summary

Several materials were found in this review. Base material for rotor shaft is not still designed, but it was supposed in the review that any structural material will need a coating to ensure enough surface properties. The antagonistic materials, the friction coefficient and wear are not described in every case.
It can be assumed that a material rubbing against itself with a high friction coefficient will produce wear on a less hard antagonistic material, but experiments showed in the past that it is not systematically the case. Every situation has to be tested. Same deposits obtained by different techniques of deposition or manufacturers may give very different results.

The 110°C operating temperature is in the problem range of temperatures for many hard coatings. Wear rates often fall off above about 300°C, even the effective temperature at the contact is perhaps much higher. With the type of contact envisaged, abrasive wear resistance would be required.

The following table aims at recommending some possible coatings for tests before choosing the final brush/rotor coating configuration.

Brush material	**Coating on rotor side**	**Associated technique of deposition**	**Wear localisation**
Low allow steel or Stainless Steel brushes ***possibly with pre-oxidation***	***Coating directly on rotor surface***		
	Thick deposit of Colmonoy	APS, PTA,TIG (D-Gun?)	Brushes
	SiC, TiC coating	PTA deposition	Brushes
	Tungsten carbide coating (with no cobalt or NiCr binder) – verify the behaviour of W-cermet WC 25%, WC_3/Cr_2C_3 70%, 5%Ni	D-Gun	Brushes
	Chromium carbide Cr_2C_3, Ni	D-Gun	Brushes
	Alumina (Al_2O_3)	VPS	Brushes
	Removable ring located around the rotor		
	Steel ring covered with hard coating	All previous possibilities	Brushes
	Ring made in Graphite mixed (or not) with MoS_2	If MoS_2, it is embedded in the graphite during the ring fabrication	Ring

As a remark, it can be noted that transfer of material from bristles to rotor will occur, interrupting smooth running. Different bristle materials have to be tested since the bristle material could either damage the rotor or contribute to a "third body" helping to lubrication.

References

[1] Deliverable D39 – HTR-E 3/12 ref 5-1-3 - Contract FIKI CT2001 - 00177

[2] XIAO, H. and T. SENDA, *Friction and wear of silicon carbide at elevated temperatures.* Journal of the Ceramic Society of Japan, 1997. **105**(9): p. 750-755.

[3] Cox, J.M. and M.G. Gee, *Hot friction testing of ceramics.* Wear, 1997. **203-204**: p. 404-417.

[4] Ouyang, J.H., S. Sasaki, and K. Umeda, *The friction and wear characteristics of low-pressure plasma-sprayed ZrO2-BaCrO4 composite coating at elevated temperatures.* Surface and Coatings Technology, 2002. **154**(2-3): p. 131-139.

[5] Ouyang, J.H. and S. Sasaki, *Microstructure and tribological characteristics of ZrO2-Y2O3 ceramic coatings deposited by laser-assisted plasma hybrid spraying.* Tribology International, 2002. **35**(4): p. 255-264.