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RAPHAEL

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DELIVERABLE (D-CT3.3)

Test program definition

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RAPHAEL ***(ReActor for Process heat, Hydrogen And ELectricity generation)***

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Document title

Test program definition

Abstract

The aim of this document is to define the test programme, in regards of past experience, and function of the lack of data for the new VHTR design.

NNC report summarises experience gained in the UK on the tribology testing useful for testing at VHTR conditions. The report will focus on the testing of graphite and consider the experience and operation in UK HTR and AGR reactor systems. The aim of this report is to provide a review of the available information and make suggestions and recommendations for the test programme proposed within RAPHAEL for the VHTR.

A common procedure is established between ANP and CEA for the test realization, and particularly the sample examinations before and after the test. Both tribometers are presented.

Two grades of graphite will be tested, like on like, representative of core / core support contact, and graphite on 800H, representative of core barrel / internals contact.

Revisions

Rev.	Date	Short description	Author	WP Leader	SP Leader	Coordinator
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RAPHAEL / SP-CT (Component DevelopmenT)

WP3: TRIBOLOGY AND CORROSION

D-CT3.3 TEST PROGRAM DEFINITION

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0	11/06/2007				Document edition

RESUME – CONCLUSIONS / ABSTRACT

The aim of this document is to define the test programme, in regards of past experience, and function of the lack of data for the new VHTR design.

A common procedure is established between ANP and CEA for the test realization, and particularly the sample examinations before and after the test.

Two grades of graphite will be tested, like on like, representative of core / core support contact, and graphite on 800H, representative of core barrel / internals contact.

SOMMAIRE / CONTENT

1. REFERENCE DOCUMENTS

[1]	HTR-E-04/04-D-5-2-1	Deliverable D40 for “Tribology in Helium Environment”
[2]	RAPHAEL-0608-D-CT3-1	Review of tribology and corrosion conditions of components for HTR and VHTR
[3]	RAPHAEL-0610-D-CT3-2	Past experience from gas cooled reactor useful for VHTR
[4]	RAPHAEL-0611-M-CT0-7	Fourth progress meeting

2. AIM OF THIS DOCUMENT

The aim of this document is to define the test programme, in regards of past experience, and function of the lack of data for the new design for VHTR.
A common procedure is established between ANP and CEA for the test realization, and particularly the sample examinations before and after the test.

3. SYNTHESIS OF FEEDBACK EXPERIENCE

The feedback experience [3] and the review of components [2] are used to define the test programme definition.

The synthesis of D-CT3.1 “Review of tribology and corrosion conditions of components for HTR and VHTR” can be synthesized by graphs in Figure 5 to Figure 7. The figures give for each component, and for a type of solicitation, the level of contact load and the temperature range.

Then for each contact situation, a table was completed during last progress meeting [4] with column past experience (see Table 2). This review help us to define if there is a miss of data and what is interesting to test now, knowing that the number of tests is limited due to the budget and the difficulty to perform a test at high temperature with an accurate environment.

4. TESTS PROGRAM DEFINITION

Considering the remarks of last paragraph, the Table 3 has been established during last meeting [4] where all partners were represented. It has been assumed that a lack of data was the tribological behaviour of new graphite grades. It’s not the alone lack of data, but it’s important to improve the knowledge of that new materials.

5. CONDITIONS OF TESTS

5.1. GAS COMPOSITION

The recommendation from partners for the level of impurities in helium gas is indicated in Table 1:

Impurity	Concentration in vpm	Partial pressure for 50 bar in µbar
H_2O	< 0.1	< 5
CO	< 2	< 100
N_2	< 1	< 50
H_2	< 5	< 250
CH_4	< 1	< 50

Table 1: gas composition recommendation issued from HTR-E

CEA and ANP will take into account the partial pressure in µbars to be representative from the reactor condition.

Only one gas composition will be tested during these test campaign.

5.2. MATERIALS

The materials are issued from SP-ML, especially for graphite grades (see Table 3: test list). Graphite 1 is identified, and Graphite 2 can be one of the two propositions.
The samples will present fresh surfaces (without pre-oxidation). Alone the temperature rising time will eventually modify the surface by corrosion reactions.

5.3. TYPE OF MOTION

The displacement will be continuing during the test (without latency time).
Each tribometer has an alternative displacement.
The velocity will be stable on a value as near as possible of 80% of the cycle. The displacement signal is an adapted triangular signal.
The stable velocity value is 1 cm/s for all tests.

5.4. CONTACT PRESSURE

The contact pressure varies from 5 to 20 MPa. These levels of pressure might be adapted in regard to the results of first tests.

5.5. COVERED LENGTH

The total covered length will be 200 m. The test can be stopped to estimate the level of degradation if needed, and pursued after.

6. TRIBOMETERS DESCRIPTION

6.1. CEA

See CEA document number DTN/STPA/LTCG 2007-008.

6.2. AREVA NP

URANIE allows the study of components subject to fretting or sliding solicitations. The internal surfaces of the tribometer (in contact with the gas and at high temperature) are not reactive with the HTR typical environment. A functional diagram is given on Figure 1. The gas process connected to the tribometer is presented in Figure 2. Test samples and the tribometer are shown in Figure 3 and Figure 4.

The main performances are as following:

- Temperature up to 1200°C;
- Pressure: atmosphere + ε;
- Helium + impurities environment;
- Alternative movement (± 9 mm) or fretting (amplitude from ±10 to ±200 µm, frequency up to 100 Hz);
- Contact pressure from 0.5 to 1000 MPa (plane on plane or pin on disk);
- Measurements: normal force, tangential force, wear, temperature.

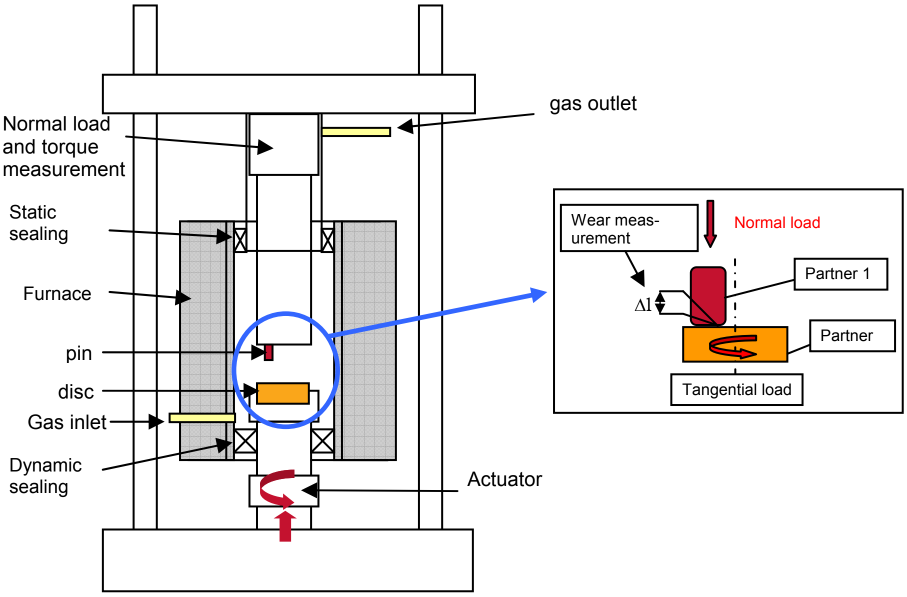


Figure 1: URANIE – AREVA NP high temperature tribometer – scheme

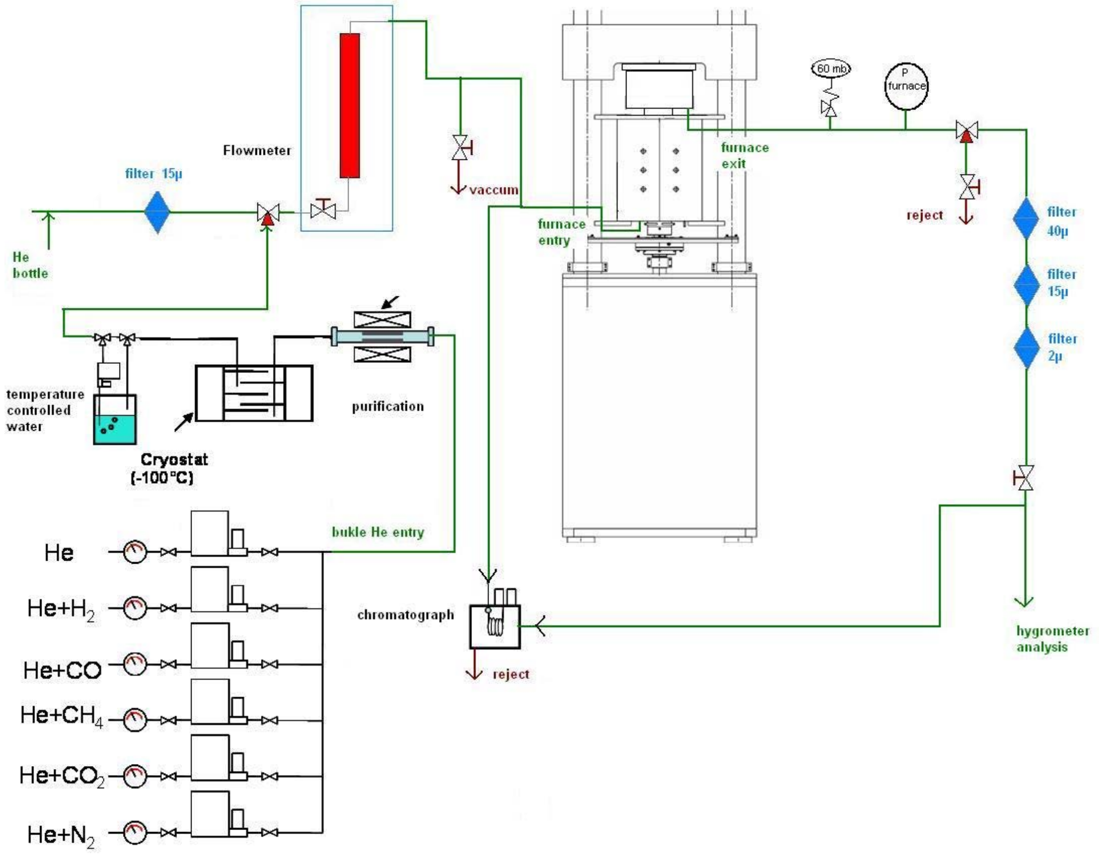


Figure 2: He + impurities process scheme

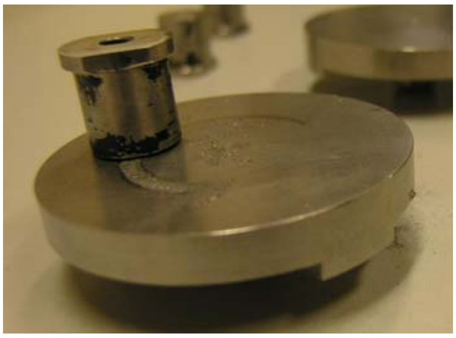


Figure 3: samples (pin on disk)

Figure 4: URANIE - Tribometer picture

7. COMMUN PROCEDURE

The test protocol has to be the same whatever the used tribometer or the type of realized test. The procedure has been improved following HTR-E feedback experience.

7.1. Sample preparation

Bulk: material report to have the chemical composition, mechanical and hardness characteristics.

Surface finish: Ra ≤ 0.8 for metallic materials (800H)
For graphite, no specification (a few µm). To be measured

Cleanliness: Alcohol + ultrasonic cleaning for metallic samples;
Dry representative surface required

7.2. Sample assessment

Visual examination:

- Identification of the rubbing surface
- Sample colour
- Picture of the entire sample before cleaning
- Sample of the debris (morphology)
- Cleaning: dry air + paintbrush
- Picture after cleaning

Worn volume measurement (weight loss, prolilometry, …)

Options:

- SEM and EDS analysis;
- Hardness and micro-hardness on cross section.

7.3. Gas control

Control of the evolution of the gas composition during the test (at least at the beginning of the test, at the middle, and at the end).

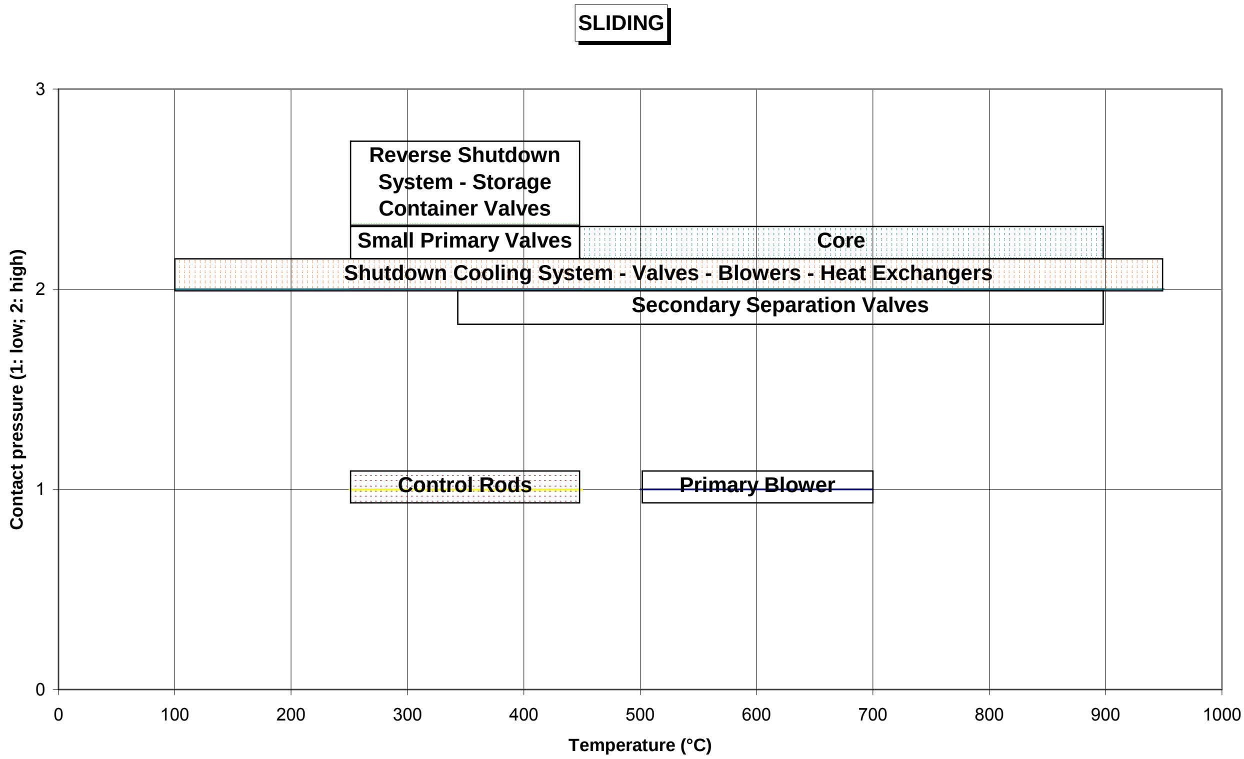


Figure 5: component working range - Sliding

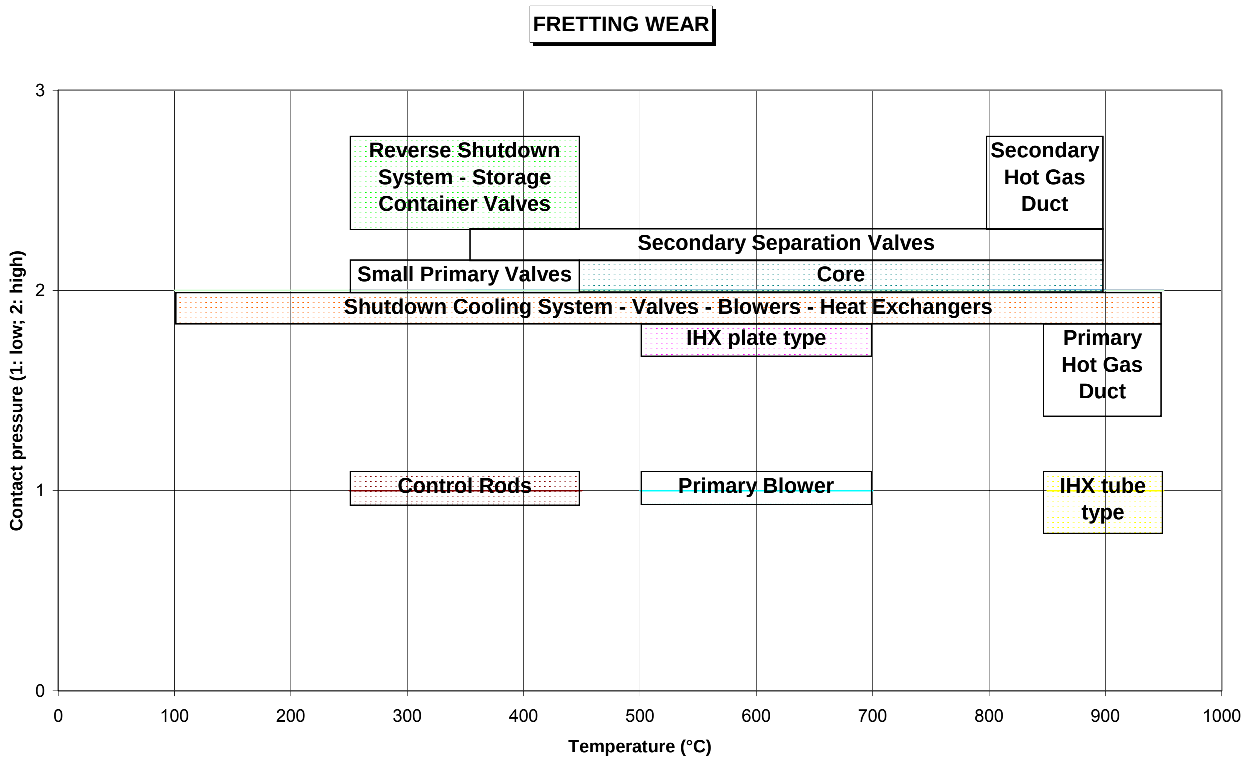


Figure 6: component working range - Fretting

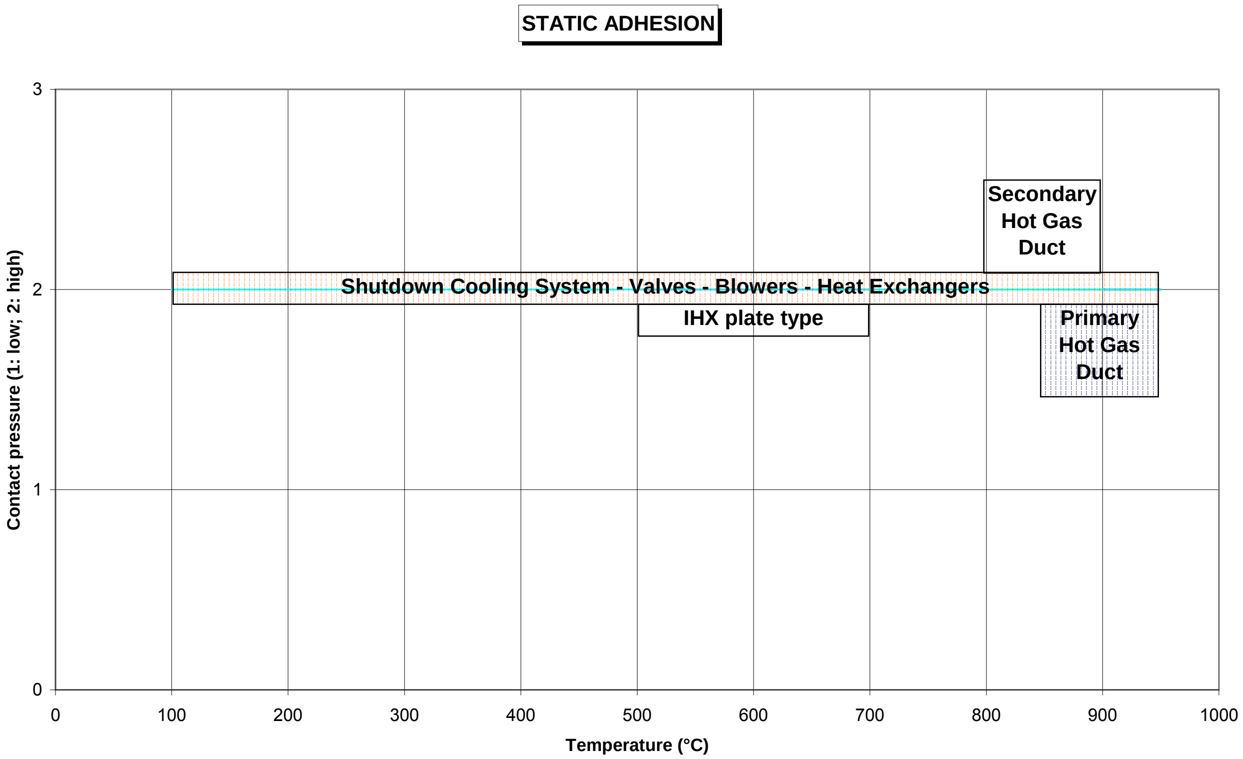


Figure 7: component working range – static adhesion

Table 2: Synthesis of feedback experience in regards of needs

Component	Area	Tempera-ture	Type of Motion	Contact pressure	Relative displace-ment speed or frequency	Possible Materials	Potential Prob-lems	Past experi-ence
Primary Hot Gas Duct	Liner	850 - 950°C	Thermal Movement Vibration	H	S	Alloy 800 or Alloy 617	Static adhe-sion/Sticking Fretting Long life	Done ZrO2/Y203(13%vol)+NiCrAlY
Heat Exchanger Plate Type	Plate supports	500 - 700°C	Thermal Movement Vibration	H	S	Alloy 800 or Alloy 617 or Alloy 230	Static adhe-sion/Sticking Fretting Long life	No past experience
Heat Exchanger Tubular Type	Tube spacers	850 - 950°C	Thermal Movement Vibration	L	F	Alloy 800 or Alloy 617 or Alloy 230	Static adhe-sion/Sticking Fretting Fatigue Long life	Done except with 230
Primary Blower	Catcher bearings	< 100°C Accidental condition TBD	Sliding	H	S	TBD Ceramic balls? SiC?	High speed Long life Fretting	Partial past experi-ence New tests
Small Primary Valves	Sealing systems Guiding system Drive mechanism	250 - 450°C	Sliding	H	S	TBD	High speed Long life Fretting Fatigue	Done

Component	Area	Tempera-ture	Type of Motion	Contact pressure	Relative displace-ment speed or frequency	Possible Materials	Potential Prob-lems	Past experi-ence
							Gas tightness	
Control Rods	Drive mechanism	250 - 450°C	Sliding	L	S	TBD	Low speed Long life Fretting Fatigue	Need of more in-formation about the design
Core	Blocks Bricks Keys Supports Core barrel	450 - 900°C Accidental condition TBD	Thermal movement Sliding	H	S	Graphite (Composite strip NBG17, 18, PCEA) Type 316 stainless steel 9Cr, 800, 718	Brick restraint Brick movement Thermal cycle wear Cracking from keyed slots	New graphite grade (with good oxidation behav-iour) No experience
Secondary Hot Gas Duct	Liner	800 - 900°C	Thermal movement Vibration	H	S	Alloy 800 or Alloy 617	Static adhe-sion/Sticking Fretting Long life	Later
Secondary Separa-tion Valves	Sealing system Guiding system Drive mechanism	350 - 900°C	Sliding	H	S	TBD	High speed Fretting Fatigue	Later

Component	Area	Tempera-ture	Type of Motion	Contact pressure	Relative displace-ment speed or frequency	Possible Materials	Potential Prob-lems	Past experi-ence
							Long life Gas tightness	
Reverse Shutdown System - Storage Container Valves	Sealing system Guiding system Drive mechanism	250 - 450°C	Sliding	H	S	TBD	High speed Long life Fretting Fatigue Gas tightness	Later
Shutdown Cooling System - Valves Blowers Heat Exchangers	Sealing system Guiding system Drive mechanism Bearings Supports	100 - 950°C	Sliding Thermal movement Vibration	H	S	TBD	High speed Long life Fretting Fatigue Gas tightness Static adhe-sion/Sticking Erosion	Need of more in-formation about the design

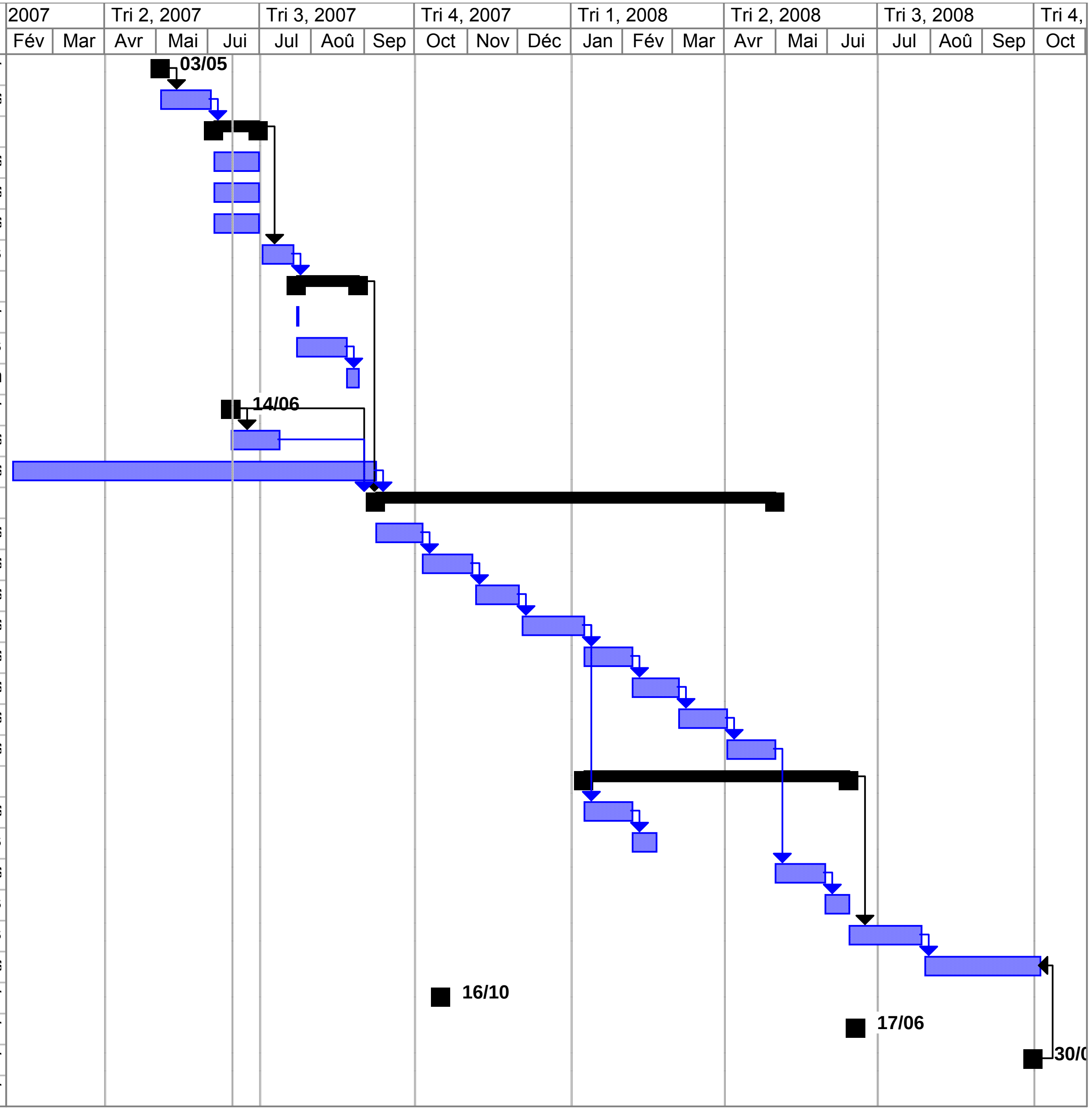
Table 3: test list

N°	Component / contact	Environment	Material 1 Disk / rail	Material 2 Pin	Temper. (°C)	Load (MPa)	Covered length (m)	Velocity (cm/s)	Tribometer ANP	Tribometer CEA	Remark
1	Core / core support	He1	Graphite 1	Graphite 1	1000	10	200	1	X	X	
2	Core / core support	He1	Graphite 1	Graphite 1	1000	20	200	1	X		High load at the bottom
3	Core / core support	He1	Graphite 1	Graphite 1	800	10	200	1	X		
4	Core / core support	He1	Graphite 1	Graphite 1	800	5	200	1		X	
5	Core / core support	He1	Graphite 2	Graphite 2	1000	10	200	1	X		
6	Core / core support	He1	Graphite 2	Graphite 2	800	10	200	1	X		
7	Core / core support	He1	Graphite 2	Graphite 2	800	5	200	1		X	
8	Core barrel / internals	He1	800H	Graphite 1	850	10	200	1	X	X	accidental condition
9	Core barrel / internals	He1	800H	Graphite 2	850	10	200	1	X		
10	Core barrel / internals	He1	800H	Graphite 1	500	10	200	1		X	Normal condition
11	Core barrel / internals	He1	800H	Graphite 2	500	10	200	1	X		

	Manufacturer	Grade	Coke	Process	Status
Graphite 1	GrafTech	PCEA	Petroleum	Extrusion	Major
Graphite 2	SGL Carbon	NBG-17 (or 18)	Pitch	Vibro-moulded	Minor (Major)

ANNEX: DETAILED TIME SCHEDULE

Nº		Nom de la tâche	Durée
1		Material definition (graphite grades)	0 jour
2		Consultation des fournisseurs	1 mois
3		**Procurements of materials**	**20 jours**
4		Graphite 1	1 mois
5		Graphite 2	1 mois
6		800H	1 mois
7		Machining	3 sms
8		**Preliminary examinations**	**15 jours**
9		Sample weight	1 jour
10		Profilometry	2 sms
11		SEM	1 sm
12		Impurity level defintion	0 jour
13		Gas procurements	1 mois
14		URANIE Qualification	7 mois
15		**Realization of tests**	**160 jours**
16		11 - 800H / NBG-17, 500°C, 1(	1 mois
17		9 - 800H / NBG-17, 850°C, 10 I	1 mois
18		8 - 800H / PCEA, 850°C, 10 M|	1 mois
19		6 - NBG-17 / NBG-17, 800°C, '	1 mois
20		3 -PCEA / PCEA, 800°C, 10 M|	1 mois
21		5 - NBG-17 / NBG-17, 1000°C,	1 mois
22		1 - PCEA / PCEA, 1000°C, 10 I	1 mois
23		2 - PCEA / PCEA, 1000°C, 20 I	1 mois
24		**Final examination**	**110 jours**
25		Profilometry - 4 first tests	1 mois
26		SEM - 4 first tests	2 sms
27		Profilometry - 4 last tests	1 mois
28		SEM - 4 first tests	2 sms
29		Data examination	6 sms
30		Report	2 mois
31		6th progress meeting - Presentation	0 jour
32		7th progress meeting - Presentation	0 jour
33		D-CT3.4 Draft last time	0 jour
34		D-CT3.4 Final version	0 jour



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RAPHAEL / SP-CT WP3 : Tribological experimental program definition.
Auteurs : L. CACHON.

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Titre	**RAPHAEL / SP-CT WP3 : Tribological experimental program definition**
Auteur(s)	L. CACHON

Résumé :
The scope of this document is the European program RAPHAEL of WP6, and more precisely the deliverable D-CT3.3. The aim of this deliverable is to define the tribological tests in an helium environment for VHTR applications.
A recall is made of the mains results of the previous deliverables, which concern tribological issues, tribological situations encountered in VHTR, the materials for tribological applications, and also the presentation of the CEA tribometer, which could simulate tribological VHTR situations. Then the document ends by a proposal of elementary tests.

Note technique vérifiée par L. AYRAULT

Mots clés	VHTR, Tribology, Graphite

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Classification				
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				X

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Révisions

Date	Objet de la révision	Indice
02/04/07	Emission initiale	0

Références :

[1] HTR-E-04/04-D-5-2-1 Deliverable D40 for “Tribology in Helium Environment”

[2] RAPHAEL-0608-D-CT3-1Review of tribology and corrosion conditions of components for HTR and VHTR

[3] RAPHAEL-0610-D-CT3-2 Past experience from gas cooled reactor useful for VHTR

[4] RAPHAEL-0611-M-CT0-7 Fourth progress meeting

Table des matières

1 INTRODUCTION

The aim of this document is to define the test programme, in regards of past experience, and function of the lack of data for the new design for VHTR.

The different parts treated in this document are listed here after :

- Materials to be tested,
- Location of the different areas which take place tribological solicitations on VHTR components.
- Existing devices to be used for tests, with their characteristics.
- Experimental protocol to lead tribological tests.
- Main representative elementary tests.

2 TRIBOLOGICAL SOLICITATIONS ON VHTR COMPONENTS

Based on the document [2] which is a review of tribological operating conditions of the main components, the table in appendix 1 is a synthesis these tribological solicitations.

The main components are :

- Valves : for inventory circuit or cold part of principal circuit, the temperature never goes up to 400°C. The maximum contact pressure of a such component is about 20 to 30 MPa. For GCR concepts and especially indirect cycle, valves are needed to cut the hot gas flow (up to 1000°C).
- Blower : for bearings or catcher bearings.
- Seals : several seals are located to the tightness of the different stage of the stator. Seal is needed too, for the thermal expansion unit of the hot duct.
- Control Rod Drive Mechanism (CRDM) for several parts : first the guidance of the rack between gears, and second, the fuel entrance.
- Internals/core : two part are distinguished. The contact between internals and the structure and the contact between internal themselves.
- Bearings.

For each tribological solicitation, pressure contact and temperature are listed.

It appears that the most critical situations, to be studied in priority, concern the core, especially the graphite / graphite contact and also the metal (structure of the vessel) /graphite contact.

3 MATERIALS

The materials tested are different grade of graphite, able to be used in VHTR. NNC who is in charge of the RAPHAEL task SP-ML, has to specify two different grades of graphite.

4 TRIBOMETER FOR TESTS : CEA – HE TRIBOMETER

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

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

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

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

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

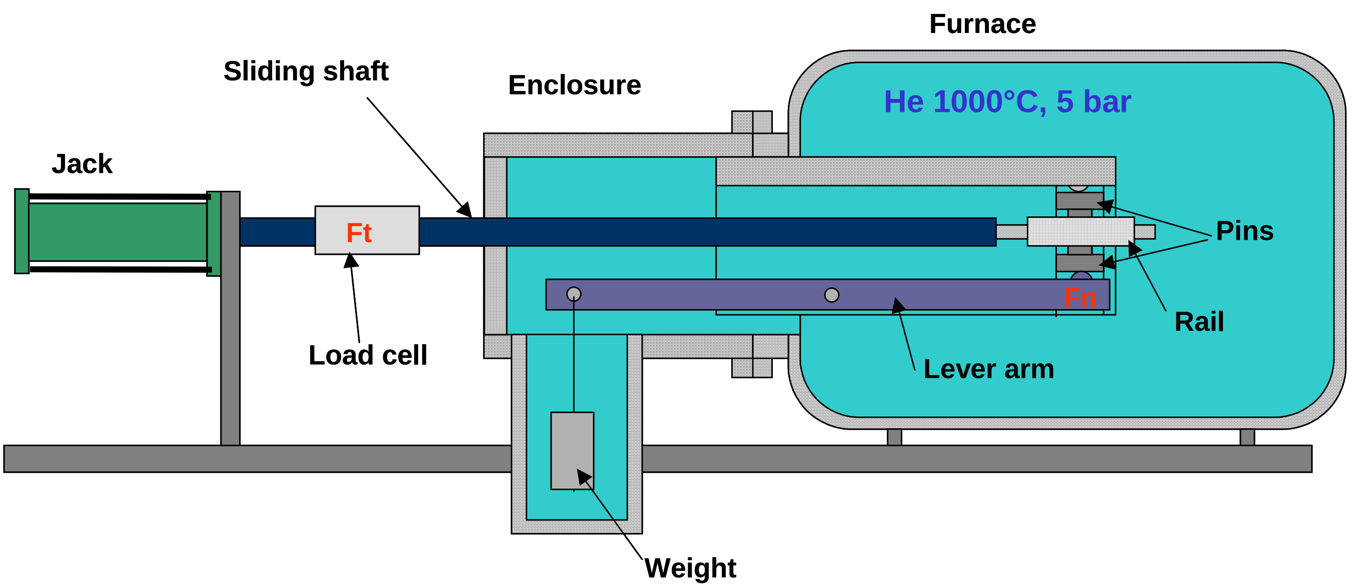


fig. 1 : schematic diagram of He tribometer.

The major characteristics of He tribometer are :

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

✓ Temperature : up to 1000°C,Steam, impuritiesPhotographies of samples and He tribometer can be seen in appendix 2.

5 TEST PROGRAM DEFINITION

Taking into account the critical issue of the contact behaviour of the graphite (§3), in the core vessel, an experimental program (table 1) were proposed [4] taking into account the common interest of each partners and also the limited number of tests.

N°	Component / contact	Environment	Material 1 Disk / rail	Material 2 Pin	Temper. (°C)	Load (MPa)	Covered length (m)	Velocity (cm/s)	Tribometer FANP	Tribometer CEA	Remark
1	Core / core support	He1	Graphite 1	Graphite 1	1000	10	200	1	X	X	
2	Core / core support	He1	Graphite 1	Graphite 1	1000	20	200	1	X		High load at the bottom
3	Core / core support	He1	Graphite 1	Graphite 1	800	10	200	1	X		
4	Core / core support	He1	Graphite 1	Graphite 1	800	5	200	1		X	
5	Core / core support	He1	Graphite 2	Graphite 2	1000	10	200	1	X		
6	Core / core support	He1	Graphite 2	Graphite 2	800	10	200	1	X		
7	Core / core support	He1	Graphite 2	Graphite 2	800	5	200	1		X	
8	Core barrel / internals	He1	800H	Graphite 1	850	10	200	1	X	X	accidental condition
9	Core barrel / internals	He1	800H	Graphite 2	850	10	200	1	X		
10	Core barrel / internals	He1	800H	Graphite 1	500	10	200	1		X	Normal condition
11	Core barrel / internals	He1	800H	Graphite 2	500	10	200	1	X		

Table 1 : Main tribological program conditions.

CEA will be in charge of 5 tests. Three of them are devoted to homogeneous graphite contact (2 grades tested) at two level of temperature (1000°C and 800°C) and two level of loads (10 and 5 MPa). The 2 others simulate the contact between the metallic components and the graphite core. So heterogeneous contact between 800 H alloy and graphite will have to be simulated, at two levels of temperature (850°C and 500°C) and with pressure load of 10 MPa.

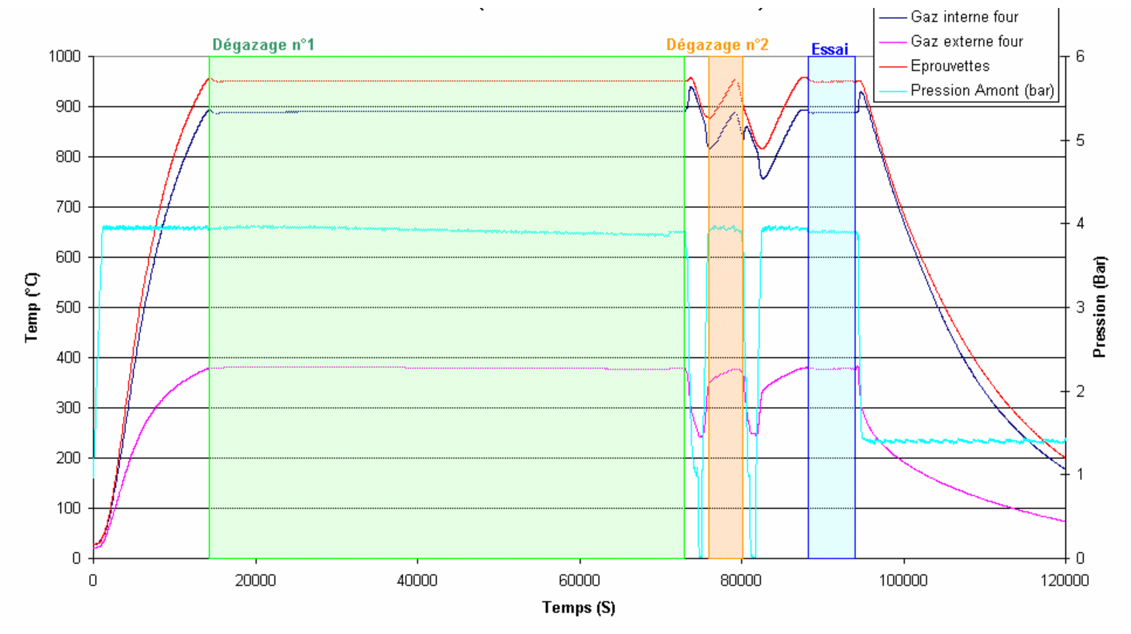
5.1 Gas composition

The recommendation from partners are indicated in the table 2 :

	µbar	Ppm for 3bar
H_2O	<7	<3
CO	<140	<47
N_2	<70	<24
H_2	<350	<117
CH_4	<70	<24

Table 2 : Gas composition

To obtain these high values of impurities, specific procedure to prepare the tribometer has to be carried out. It consists of two degassing of the furnace at a temperature higher than the running temperature following by vacuum pumping. It allows to have a level of impurities lower than the specifications (figure 2).



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

Figure 2 : Illustration of the procedure to have an Helium atmosphere conform to the specifications.

5.2 Type of motion

As it is explained in §4, the CEA tribometer generates alternative motion. The amplitude of the motion is 20 mm and so, one cycle represents 40 mm.

To obtain 200m, 5000 cycles will have to be done, without stop or removal for intermediate analyses. Only if the degradations of the sample not allow to continue tests that they will be stopped.

The velocity is 1 cm/s for all tests and is stable on 80% of the cycle. The displacement signal is triangular (figure 3).

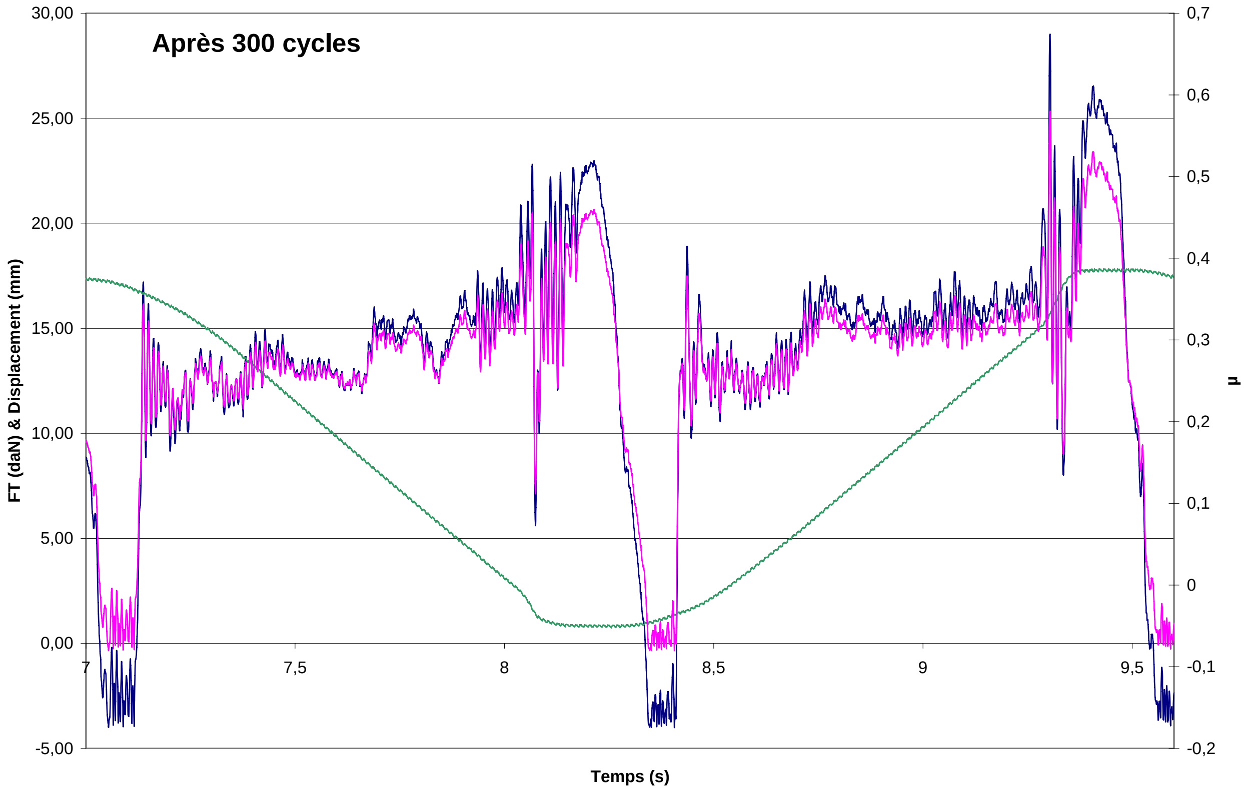


Figure 3 : Illustration of the differents signal recorded during the tests.

5.3 Test protocol

The test protocol has to be the same between CEA and AREVA –NP to obtain comparable results, even if the two tribometers are different. The following procedure has been improved during HTR-E feedback experience.

5.3.1 <u>Sample preparation</u>

Massive bodies:

- Material report, to have the chemical composition, mechanical and hardness characteristics.

- o Same supplier of samples (if possible)

Surface finish:

- o Ra < 0.8 for metallic materials (800H)
- o For graphite, specifications have to be précised by NNC.

Cleanliness:

- o Alcohol + ultrasonic cleaning for metallic samples;
- o For graphite, specifications have to be precised by NNC.

5.3.2 Sample assessment

Visual examination:

- o Identification of the rubbing surface
- o Sample colour
- o Picture of the entire sample before cleaning
- o Sample of the debris (morphology)
- o Cleaning: dry air + paintbrush
- o Picture after cleaning
- o Worn volume measurement (weight loss, profilometric method, …)

Options:

- o SEM and EDS analysis;
- o Hardness and micro-hardness on cross section.

6 CONCLUSIONS

After a recall of the mains results of the previous deliverables, which concern tribological issues, tribological situations encountered in VHTR, materials for tribological applications, and also the presentation of the CEA tribometer which could simulate tribological VHTR situations, the document proposes elementary tests.

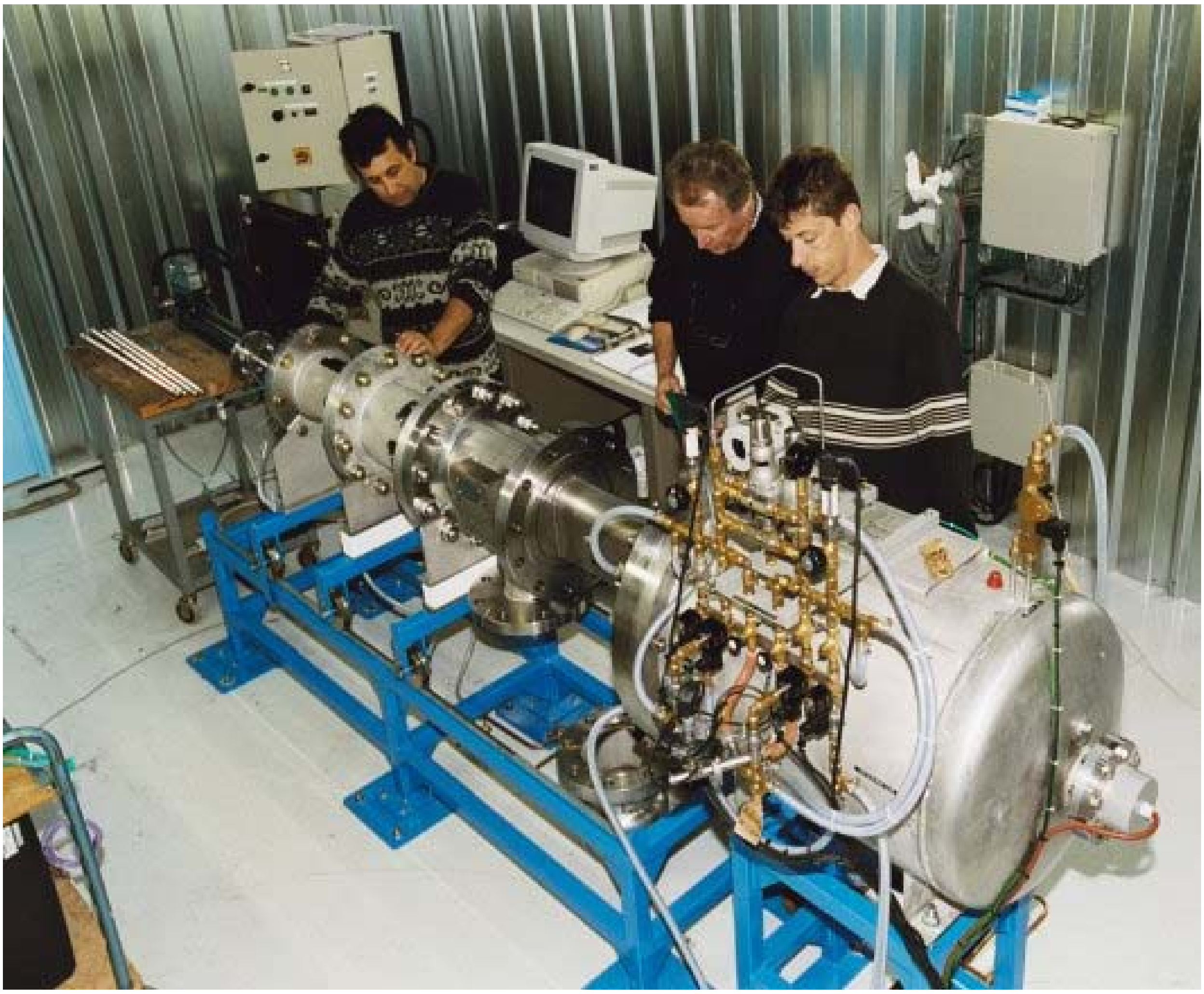
APPENDICES

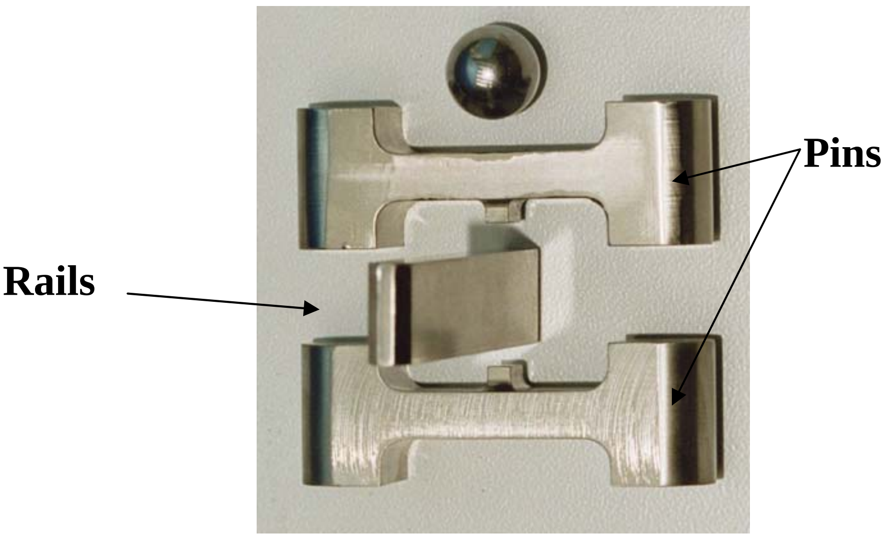
7.1 Appendix 1 : Tribological situations in a VHTR

Component	Area	Temperature	Type of Motion	Contact pressure	Relative displacement speed or frequency	Possible Materials	Potential Problems	Past experience
Primary Hot Gas Duct	Liner	850 - 950°C	Thermal Movement Vibration	H	S	Alloy 800 or Alloy 617	Static adhesion/Sticking Fretting Long life	Done ZrO2/Y203(13%vol)+NiCrAlY
Heat Exchanger Plate Type	Plate supports	500 - 700°C	Thermal Movement Vibration	H	S	Alloy 800 or Alloy 617 or Alloy 230	Static adhesion/Sticking Fretting Long life	No past experience
Heat Exchanger Tubular Type	Tube spacers	850 - 950°C	Thermal Movement Vibration	L	F	Alloy 800 or Alloy 617 or Alloy 230	Static adhesion/Sticking Fretting Fatigue Long life	Done except with 230
Primary Blower	Catcher bearings	< 100°C Accidental condition TBD	Sliding	H	S	TBD Ceramic balls? SiC?	High speed Long life Fretting	Partial past experience New tests
Small Primary Valves	Sealing systems Guiding system Drive mechanism	250 - 450°C	Sliding	H	S	TBD	High speed Long life Fretting Fatigue Gas tightness	Done
Control Rods	Drive mechanism	250 - 450°C	Sliding	L	S	TBD	Low speed Long life Fretting Fatigue	Need of more information about the design
Core	Blocks Bricks Keys Supports Core barrel	450 - 900°C Accidental condition TBD	Thermal movement Sliding	H	S	Graphite (Composite strip NBG17, 18, PCEA) Type 316 stainless steel 9Cr, 800, 718	Brick restraint Brick movement Thermal cycle wear Cracking from keyed slots	New graphite grade (with good oxidation behaviour) No experience
Secondary Hot Gas Duct	Liner	800 - 900°C	Thermal movement Vibration	H	S	Alloy 800 or Alloy 617	Static adhesion/Sticking Fretting Long life	Later
Secondary Separation Valves	Sealing system Guiding system Drive mechanism	350 - 900°C	Sliding	H	S	TBD	High speed Fretting Fatigue Long life Gas tightness	Later
Reverse Shutdown System - Storage Container Valves	Sealing system Guiding system Drive mechanism	250 - 450°C	Sliding	H	S	TBD	High speed Long life Fretting Fatigue	Later

Component	Area	Temperature	Type of Motion	Contact pressure	Relative displacement speed or frequency	Possible Materials	Potential Problems	Past experience
							Gas tightness	
Shutdown Cooling System - Valves Blowers Heat Exchangers	Sealing system Guiding system Drive mechanism Bearings Supports	100 - 950°C	Sliding Thermal movement Vibration	H	S	TBD	High speed Long life Fretting Fatigue Gas tightness Static adhesion/Sticking Erosion	Need of more information about the design

7.2 Appendix 2 : CEA – He tribometer.





RAPHAEL SP CT WP3-
VHTR tribology test program definition - experience from NNC test facilities and tests

B F Riley

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1.0 INTRODUCTION

This report summarises experience gained in the UK on the tribology testing useful for testing at VHTR conditions. The report will focus on the testing of graphite and consider the experience and operation in UK HTR and AGR reactor systems. UK experience in the tribology of graphite in helium stems largely from the Dragon Project and test programmes in support of a Mk III gas-cooled reactor. Experience of tribology in gaseous environments was also gained from the operation of the Magnox and AGR reactors, and from the test programmes supporting them.

The aim of this report is to provide a review of the available information and make suggestions and recommendations for the test programme proposed within RAPHAEL for the VHTR.

This document contains the contribution from NNC towards Deliverable D-CT3.3 of the RAPHAEL Project.

2.0 TESTS FOR DRAGON AND HELIUM-COOLED HTR AND FOR AGR

2.1 Introduction

Frictional forces and wear (adhesive and fretting) in a gas-cooled reactor can occur as a consequence of the insertion and withdrawal of reactor components and fuel as they contact the graphite core. The adhesion of stationary contacting surfaces such as those associated with the graphite core blocks can also give rise to problems. For the graphitic materials adhesive wear, and friction were recognised as important concerns on the Dragon and AGR Projects and some specific test work was carried out to assess behaviour

2.2 Tests carried out for the Dragon & AGR

During the Dragon Project, Static Adhesion, friction and wear testing were carried out in 'pure' and 'impure' helium at temperatures of up to 800°C. Friction and wear tests were carried out in a reciprocating sliding configuration in pure helium at gas pressures of 1, 20 and 50 bar. Tests in reactor quality helium with controlled impurity levels representative of a boiler leak condition were carried out at a gas pressure of 20 bar. The tests were performed in circulating helium, the gas being purified in a helium purification plant. Tests in CO_2 in support of the AGR programme were broadly in line with those from Dragon. Sections 3.0 to 5.0 below review the tests performed to address the issues concerning static adhesion, friction, wear and fretting for graphite to graphite and graphite to metal pairings.

3.0 STATIC ADHESION

The static adhesion of candidate materials for a UK helium-cooled reactor system was investigated during the early 1970s (Ref 1). Tests were carried out on stacks of metallic and other specimens at temperatures in the range 400 – 800°C. Graphite was included in the tests. 'Pure' (< 2 μ-atmospheres water) and 'impure' (50 μ-atmospheres water) helium atmospheres were employed, at pressures of 1.24 bar and 42.4 bar. The interface pressure was 0.3 MPa.

Graphite (Gilsocarbon) reacted with, but did not adhere to, both En 58J stainless steel and Nimonic 75 at temperatures of 650°C and above, in tests in impure gas at both gas pressures. Graphite-graphite couples did not adhere in any of the tests.

4.0 FRICTION AND WEAR

4.1 Programme 1 – graphite/graphite and graphite/metal pairs

In the UK during the 1960s and 1970s, research was carried out into the friction and wear of pile grade and bearing graphites, in graphite-graphite and graphite-metal couples (Ref 2). The tribological properties of these materials in low pressure CO_2 and helium, each gas containing <10 vpm water vapour, were determined at temperatures between 20°C and 600°C. Specific wear rates and friction coefficients were determined in a crossed-cylinders configuration. Three bearing grades of graphite were rubbed against several alloys, including a nitrided steel (LK5) and an austenitic stainless steel (En 58B). A pile grade graphite was also tested, but, in the crossed-cylinders configuration, in carbon dioxide only. The tests were performed under a load of 22 N.

In helium, the wear rate vs temperature graphs were similar for the three bearing graphites in tests against nitrided steel, as shown in Figure 1. The specific wear rates of the graphites increased markedly above 400°C. The friction coefficients were low, at 0.1 – 0.4, and showed little systematic dependence on temperature. Graphite wore to a greater degree against austenitic stainless steel, as shown in Figure 2. One grade of graphite (EY 105) wore severely. EY 105 was a nonmetallic-impregnated electro-graphite. A similar effect was seen for the same combination of materials in the carbon dioxide tests, but to a lesser degree. In general, friction and wear behaviour in helium was more diverse than in carbon dioxide. Pile grade graphite was subject to mild wear (~10^{-15} m^3/Nm) at all test temperatures in carbon dioxide, and gave rise to low values of μ (0.25 at 20°C, decreasing to 0.15 at 575°C).

4.2 Programme 2 – graphite/graphite pairs

Tests were carried out in 'pure' and reactor helium at temperatures from 20°C to 800°C (Ref 3). A large programme of work was carried out on several materials couples under specified conditions of contact pressure, gas pressure and temperature. Graphite (Gilsocarbon), a fine grained isotropic material, R_a = 2 – 5 µm was tested in a like-on-like configuration. The object of the testing was to determine

A. whether there is any significant difference between friction and wear performance in pure helium and in helium representative of gas composition in a reactor under water leakage conditions.

B. whether gas pressure affects friction and wear behaviour in pure helium.

The gases used were denoted ‘pure’ or ‘reactor gas’. Tests in ‘pure’ gas were carried out at 1 bar and 50 bar. Reactor gas, at 20 bar, contained 25 vpm hydrogen, 25 vpm carbon monoxide, 2.5 vpm carbon dioxide and 2.5 vpm water. In each test, two pairs of discs, 12.7 mm and 25.4 mm in diameter, were mounted one above the other and rubbed simultaneously, the two specimens being subjected to equal contact pressures, 2.1 MPa or 6.5 MPa.

Some results from the graphite tests are presented in Figures 3 to 7. In these tests, the friction coefficient seldom exceeded 0.4. Wear was low, and independent of temperature and contact pressure in the range 300 – 800°C. At 20°C, especially at high contact pressures, the wear rate was high. There was no significant effect of gas pressure on either friction or wear in the graphite tests at elevated temperatures.

The following conclusions were drawn from this test programme:

a. No significant or systematic difference was observed between wear behaviour in pure helium and in reactor gas.

b. In pure helium, no systematic effect of gas pressure was observed.

c. Friction and wear show some dependence on temperature, as described above.

d. There was no systematic effect of contact pressure on friction.

e. The water and oxygen content of the helium had considerable influence on the friction of steels.

f. Graphite (Gilsocarbon) gave good results in all tests at temperatures in the range 400°C to 800°C but wore rapidly at 20°C.

4.3 Programme 3 – graphite/metal and graphite/graphite pairs

A large number of materials couples with potential for use in a UK High Temperature Reactor were tested in a flat-on-cylinder arrangement in low pressure helium at 700°C (Ref 4). The level of water in the apparatus was < 2 vpm.

In general, structural alloys behaved poorly in pure helium at elevated temperature, adhesive transfer being a feature of most tests. However, the results for graphite (gilsocarbon) were good, particularly in a like-on-like arrangement. This combination was the subject of further tests at temperatures from 20°C to 800°C, before and after degassing the apparatus to reduce the impurity levels in the gas. The results are presented in Table 2. Degassing before test clearly had an effect on the tests at 20°C, the wear rate increasing with the purity of the gas. For the elevated temperature tests, the situation was less obvious.

4.4 Programme 4 - the effect of dwells on metal/graphite pairs

In a High Temperature Reactor, some surfaces will rub together during temperature cycles or when components are inserted or withdrawn from the reactor. In order to assess the effect of dwells between such operations, a series of plate-on-disc reciprocating motion tests was performed in low pressure (1.68 bar) helium at 450°C, 650°C and 750°C using several materials combinations (Ref 5). Graphite (EYC 9106) was tested against 316 stainless steel. The tests were performed in a controlled atmosphere containing 5 µatmospheres water. The tests constituted either 100 continuous cycles, or 5 cycles per day for 20 days, under a contact pressure of 6.5 MPa. All tests were duplicated.

The friction coefficients in the 316/graphite tests were between 0.1 and 0.5. At 450°C, µ in the dwell tests was lower than in the continuous tests. This effect was not repeated at 650°C or 750°C. The wear profiles were smooth, the plate (316) not wearing significantly. The graphite wore much more, typically at a rate of 10^{-12} m^3/Nm.

The tests showed little or no effect of dwell on either friction or wear.

5.0 FRETTING – METAL/METAL AND GRAPHITE/GRAPHITE PAIRS

A programme of impact/slide fretting tests was carried out at a gas pressure of 1.2 – 1.4 bar and temperatures of 350°C and 750°C on several materials combinations (Ref 6), including graphite (Gilsocarbon) in a like-on-like configuration.

Tests were performed in 'pure' helium, containing < 1 vpm water, and a dosed helium, containing 50 vpm water. The tests were conducted at a frequency of 75 Hz, with a slip amplitude of 0.18 mm and a vibration amplitude of 0.254 mm. The test duration was variable, mostly up to 100 hours. The contact was flat-on-flat.

The test results are presented in Tables 3 and 4. At 750°C, the wear rates found in the graphite-graphite tests fell within the range 10^{-15} – 10^{-14} m^3/Nm. The following conclusions were drawn from the study:

Effect of gas purity

For graphite combinations at high temperature a change in gas purity had little effect on the wear rate.

Effect of temperature

In graphite the damage produced at 350°C was an order of magnitude worse than that produced at 750°C.

Effect of test duration

The wear rate was constant with time in the graphite/graphite tests.

6.0 GRAPHITE

6.1 Graphite selection

A number of candidate graphites have been selected for the European HTR and are currently being irradiated in the Petten HFR as part of their qualification. These are listed in Table 5. Most have also been tested to determine their reactivity in air. It can be seen that they cover the two main coke sources (i.e. petroleum and pitch) and the main manufacturing methods (i.e. extrusion, iso-moulding and vibro-moulding). The graphites to be tested in the proposed experiments should be selected from the four major grades. If only two graphites are to be tested it is recommended that PCEA and NBG-17 (or 18) be selected in order to best cover the different coke sources/manufacturing methods. The former is a petroleum coke/extruded graphite and the latter is a pitch coke/vibro-moulded graphite.

Note - it may be useful to perform a test of graphite against the control rod material. This could be the SGL selected composite and/ or Alloy 800H.

6.2 Surface finish

For the machined graphite components used in the UK reactors (Magnox and AGRs) no surface finish specification was given. Flat surfaces will have been produced by milling and curved surfaces such as internal holes by drilling or turning (on a lathe). The surface finish following the machining operations was not subject to further operations/working to reduce any surface roughness. It is not expected that this situation would be any different for machined graphite used by any other country (e.g. US, France, Germany). The likelihood of this changing for future reactors (i.e. HTRs) is considered to be very small. Therefore it is not proposed that the specification for the graphite should include anything on the surface roughness to be achieved.

In order to get a feel for the typical surface roughness of machined graphite, two of the major graphite suppliers were asked to provide any information they had, and a brief review of available documentation has been carried out. Information received from one of the graphite suppliers is summarised in Figures 8 to 11. Figure 8 is an extract from the DIN 4762/1 standard, which defines two measures of surface roughness Ra and Rz. As graphite has an open surface structure, it is not possible to use a standard surface measuring devices and so the manufacturer has tested different methods and equipment with different surface probes using a sphere with different diameter. The two diameters chosen were 10 µm and 1.5 mm. The results for the two probe diameters are shown in Figures 9 and 10 for a milled surface and in Figure 11 for a turned surface (using a lathe). It can be seen that the surface roughness (Ra) is of the order of a few µm, and that the turned surface is the rougher of the two, which is to be expected. Information received from the other manufacturer is consistent with the above, the surface roughness for milling being ~1 µm and for turning being ~2 µm. A US report reviewed states that the graphite tested had a surface roughness of ~2.5 µm, which is consistent with the information received from the manufacturers. (Note – it is likely that the highest peaks will be removed after a small number of cycles).

7.0 CONCLUSIONS

Some conclusions with relevance to VHTR and associated testwork have been drawn from the work reported here:

- No adhesion occurred in graphite-graphite contacts at temperatures up to 800°C. There was no effect of gas pressure on the static adhesion results.
- There was no systematic effect of gas pressure on friction and wear in pure helium.
- In reactor helium at 20 bar gas pressure, friction coefficients were highly dependent upon the moisture level in the gas.
- Graphite-graphite contacts wore little in helium at temperatures in the range 400 – 800°C. The wear rates increased with increasing gas purity.
- Elevated temperature dwells resulted in more severe wear upon subsequent rubbing contact in helium.

Key issues to be considered in a future test programme include:

- The roughness of both graphite and counterface. A metallic counterface is unlikely to wear but will induce wear of the graphite, especially if the roughness of the metallic surface is not controlled to at least a normal engineering finish. A roughness of 0.8 µm will be suitable to simulate a smooth machined surface but such a surface is not smooth tribologically and will give rise to appreciable graphite wear. Tests are often carried out on surfaces with a roughness of 0.4 µm. Nevertheless, as an engineering finish 0.8 µm might be appropriate for VHTR requirements. As for the graphite surface, this should be typical of the component. Again, this is an important consideration, particularly for like-on-like tests. All specimens should be measured for roughness.

- The geometry of the metallic test specimens, especially that of pins. It is very difficult to align surfaces in flat-on-flat contact. Where one surface is unlikely to wear very much, as in graphite against an alloy surface, the flat-on-flat configuration may not become established, Large radius specimen ends can avoid the problem.

- Duplication or triplication of tests.

- How to calculate the friction coefficient systematically, allowing comparison between laboratories. There is of course a larger value at the start of each sweep and then some variation in the rest of the cycle in a reciprocating test. One could take a mean value for the central 80% of each sweep. The ranges could also be quoted but it would be good to be able to quote a single value for sliding. The mean value of the peak friction should also be measured.

- The friction and wear of graphite

If two graphites are to be tested it is recommended that PCEA and NBG-17 (or 18) be selected in order to best cover the different coke sources/manufacturing methods. The former is a petroleum coke/extruded graphite (manufactured by GrafTech) and the latter is a pitch coke/vibro-moulded graphite (manufactured by SGL Carbon). The typical surface roughness (Ra) of machined graphite (milled or turned) is of the order of a few µm. The surface finish of machined components is generally not given in a specification.

8.0 SP ML COMMENTS FOR VHTR TRIBOLOGY TESTS ON STRUCTURAL GRAPHITE

Ref	Question	SP ML Response/ comments
1	Grades of Graphite to be tested	PCEA and NBG-17 (or 18)
2	Cleaning after machining	Dry representative surface required
3	Surface preparation/ specification	Machined surface finish typical of core block (milled or turned) – all specimens measured for roughness
4	Specification of forming direction(s) in relation to the friction surface	Effect not expected to be significant

9.0 REFERENCES

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2. D F Cornelius, 'Friction and wear of some graphites rubbed against high and low chromium content alloys in carbon dioxide and helium up to 600°C', TRG Memorandum 35 (CA), 1961

3. R J Fogden, 'Friction and wear tests in helium environments', TNPG 890, 1973

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5. D Marsh, 'Friction and wear in helium atmospheres – effect of dwell', TRG Report 2732 (R/X), 1973

6. K J Campbell, 'Fretting in helium based atmospheres', BNDC R729, 1974

Table 1 Friction and wear results in pure helium at 700°C, flat-on-cylinder

Pin	Cylinder	μ_{max}	μ_{mean}	Wear rate, pin (m^3/Nm)	Wear rate, cylinder (m^3/Nm)
Gilsocarbon	Gilsocarbon	0.6 0.8	0.3 0.4	7.5E-14 5.1E-14	- -
Gilsocarbon	316	0.3 0.4	0.25 0.3	2.2E-13 2.2E-13	- -

* Pick-up (adhesive transfer)

Table 2 Friction and wear results from graphite (gilsocarbon), like-on-like, in pure helium

Temperature (°C)	Apparatus condition	μ_{max}	μ_{mean}	Pin wear rate (m^3/Nm)
20	Not degassed	0.4 0.4	0.4 0.35	1.6E-14 1.3E-14
20	2 days at 800°C	0.65 0.75	0.35 0.4	2.5E-13 2.7E-13
20	6 days at 800°C	0.65 0.7	0.3 0.35	4.3E-13 3.9E-13
400	Not degassed	0.3	0.3	2.1E-14
400	1.5 days at 800°C	1.2	0.55	3.5E-13
400	6 days at 800°C	0.85 0.7	0.4 0.35	6.0E-14 3.8E-14
700	Not degassed	1.0	0.55	8.7E-14
700	1.5 days at 800°C	0.9	0.55	4.4E-14
800	2 days at 800°C	0.5 0.55	0.3 0.3	5.0E-14 7.1E-14

Table 3 Fretting results from graphite (gilsocarbon), like-on-like, impure gas

Temp. (°C)	Freq. (Hz)	Load (N)	Test time (h)	Wear rate ($x10^{-14}$ m^3/Nm)
750	75	50	30	0.25
750	75	50	30	0.45
750	75	50	30	0.70
750	75	50	30	0.16
750	150	25	50	1.6
350	75	50	50	11
			100	12
350	150	25	50	4.2

Table 4 Fretting results from graphite (gilsocarbon), like-on-like, pure gas

Temp. (°C)	Freq. (Hz)	Load (N)	Test time (h)	Wear rate ($x10^{-14}$ m^3/Nm)
750	75	50	30	0.20
			50	0.25

Table 5 Selected graphites grades

Manufacturer	Grade	Coke	Process	Status
GrafTech	PCEA	Petroleum	Extrusion	Major
	PPEA	Pitch	Extrusion	Major
	PCIB	Petroleum	Iso-moulding	Minor
	BAN		Extrusion	Minor
SGL Carbon	NBG-10	Pitch	Extrusion	Major
	NBG-17	Pitch		Minor
	NBG-18	Pitch		Major
	NBG-20	Petroleum	Extrusion	Minor
	NBG-25	Petroleum		Minor
Toyo Tanso	IG-110	Petroleum	Iso-moulding	Minor
	IG-430	Pitch	Iso-moulding	Minor

Figure 1: Wear rates of bearing graphite in 'dry' helium

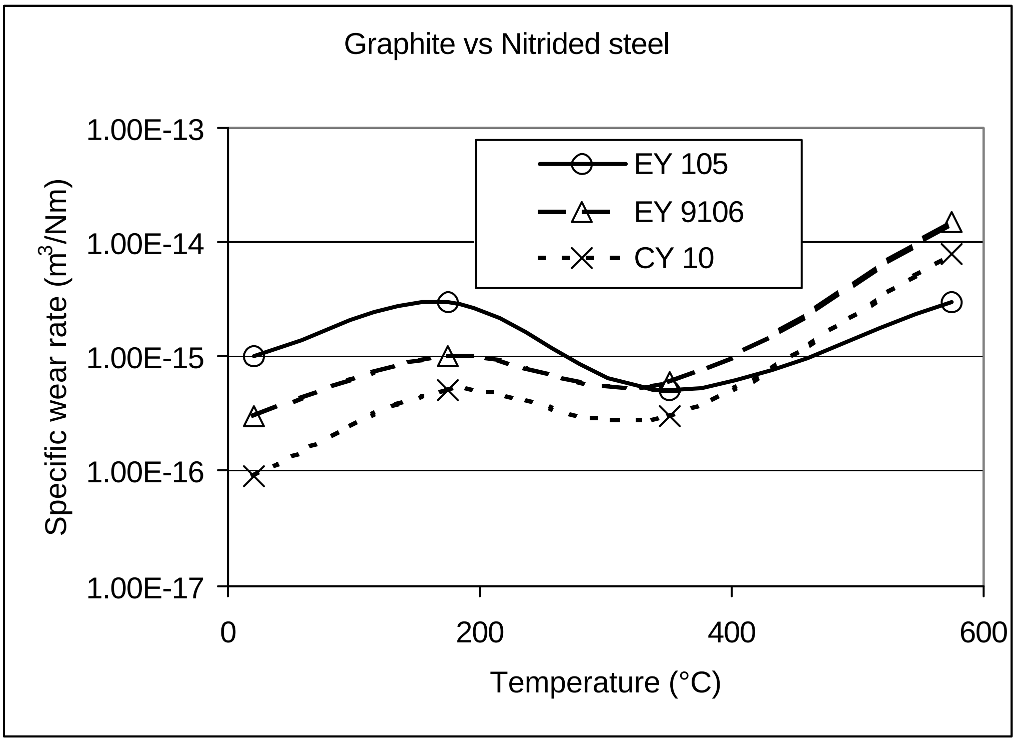


Figure 2: Wear rates of bearing graphite in 'dry' helium

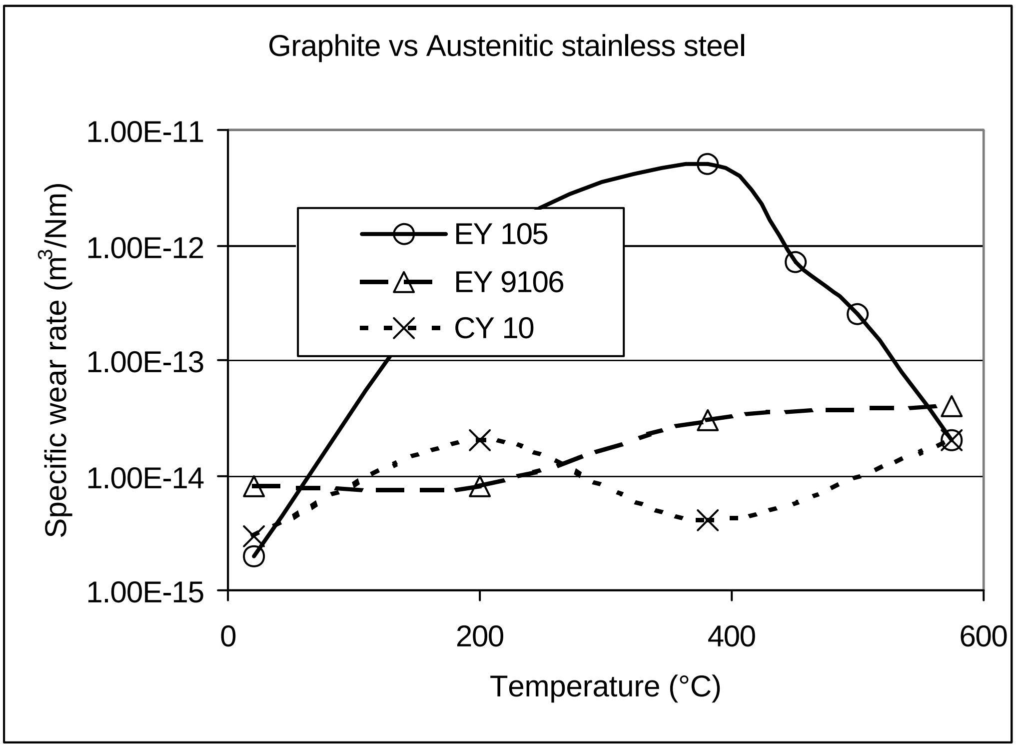


Figure 3: Effect of contact pressure on friction and wear of graphite in helium, 25.4 mm specimen

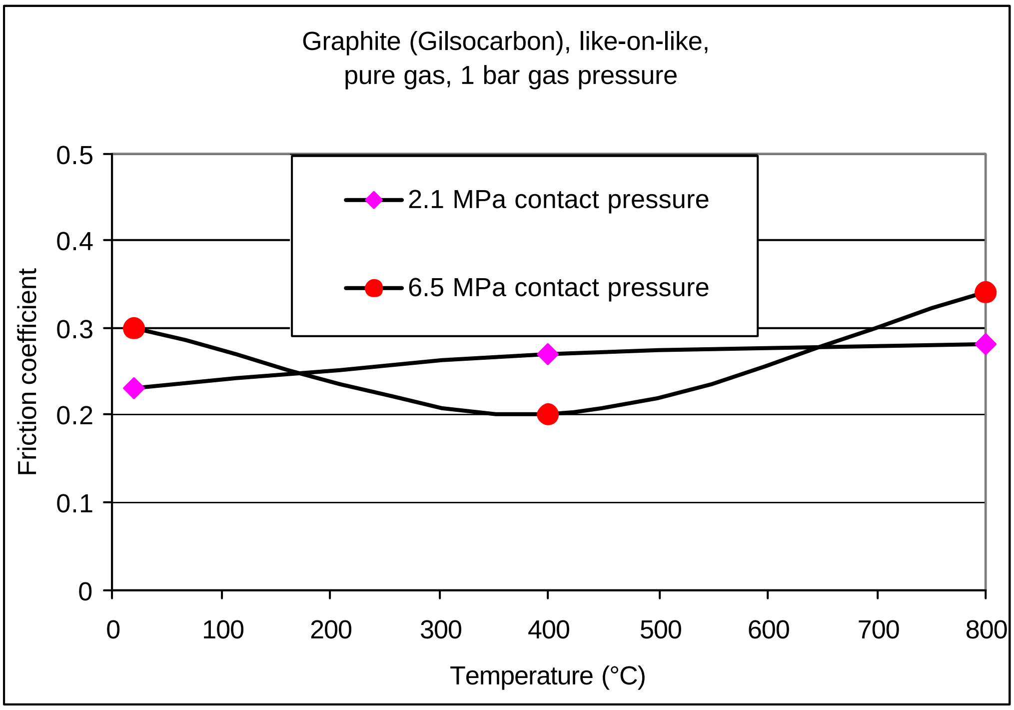


Figure 4: Effect of contact pressure on wear of graphite in helium, 25.4 mm specimen

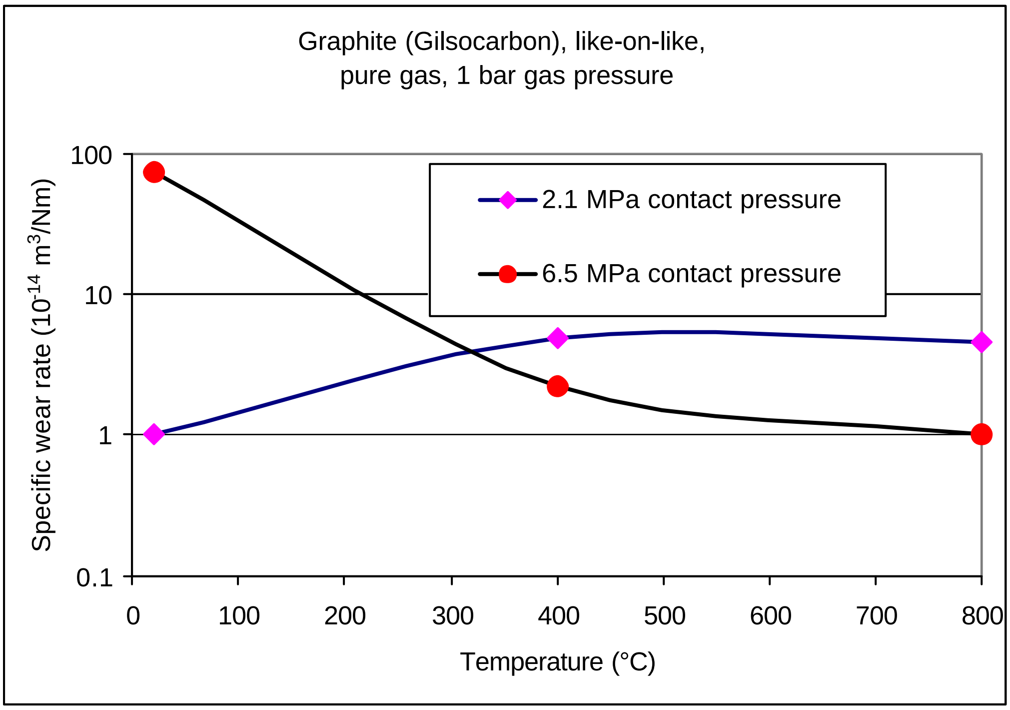


Figure 5: Effect of gas pressure on friction of graphite in helium, 25.4 mm specimen

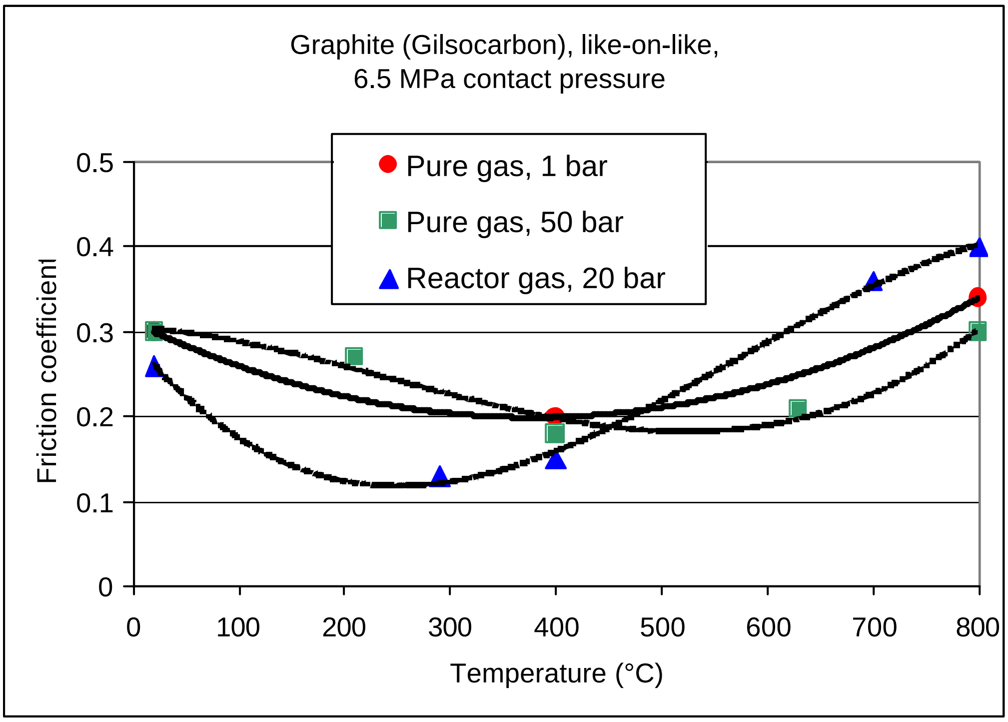


Figure 6: Effect of gas pressure on wear of graphite in helium, 25.4 mm specimen

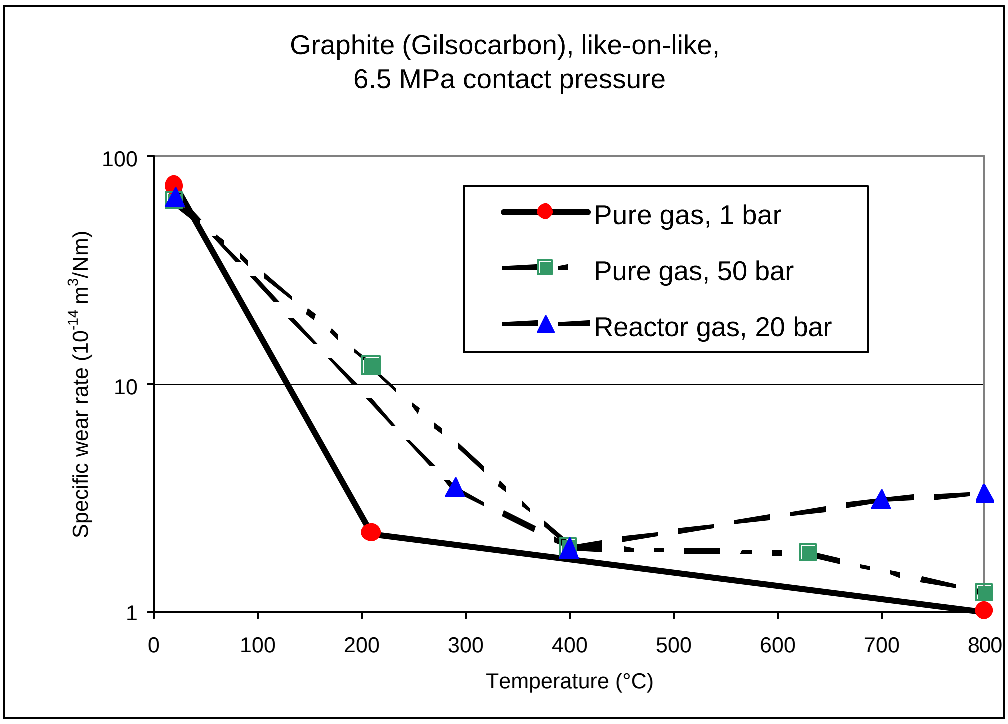


Figure 7: Effect of gas pressure on wear of graphite in helium, 12.7 mm specimen

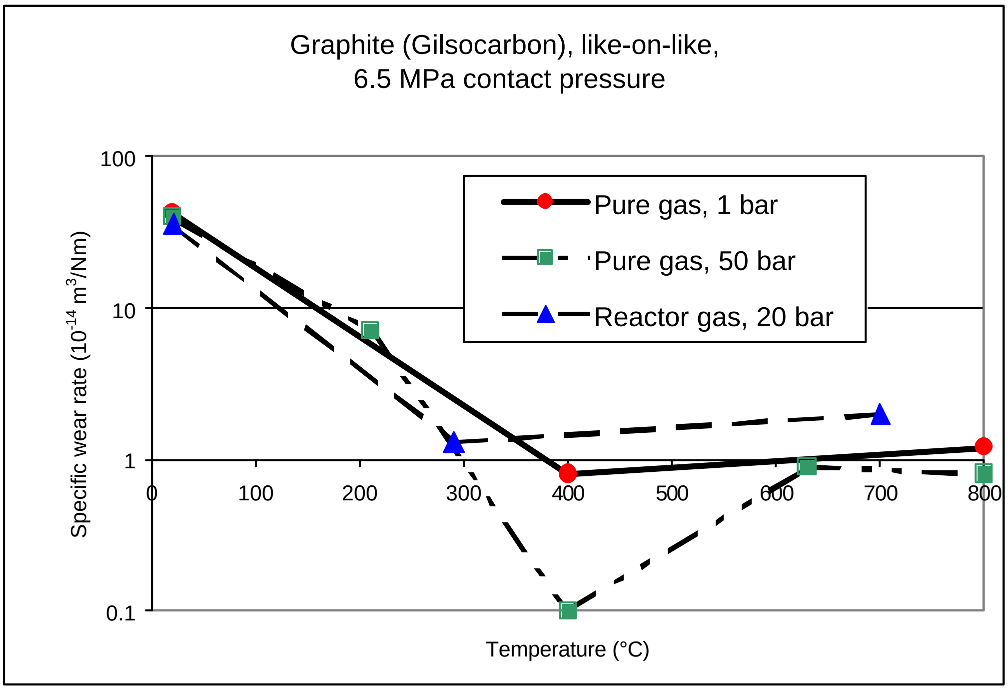
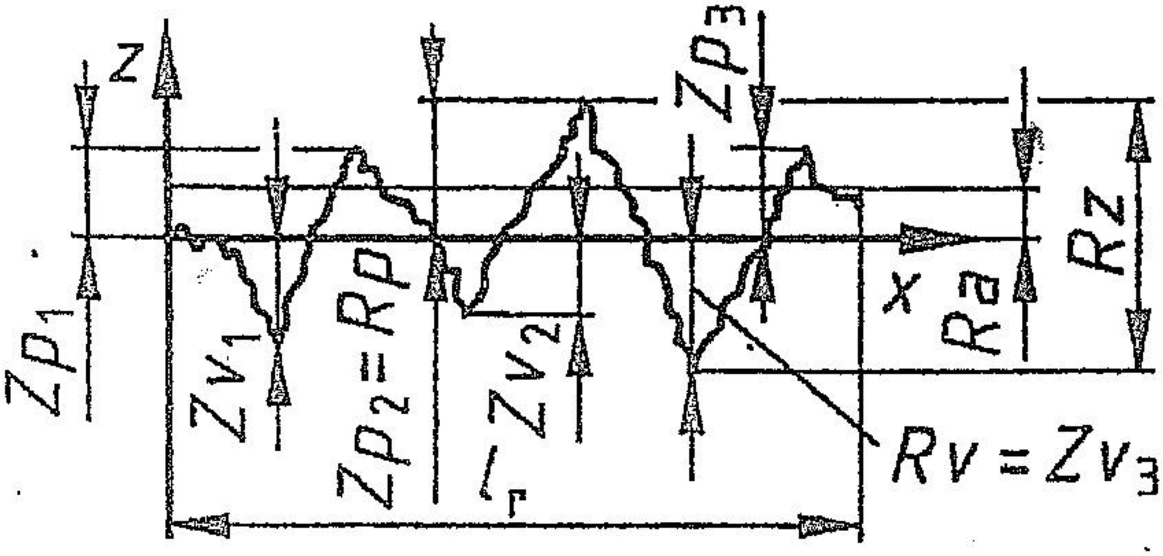


Figure 8: Extract from DIN standard

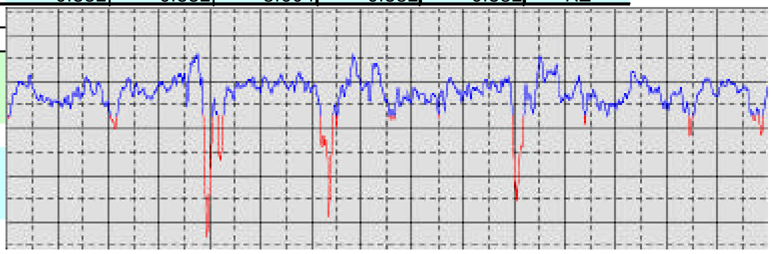


Where

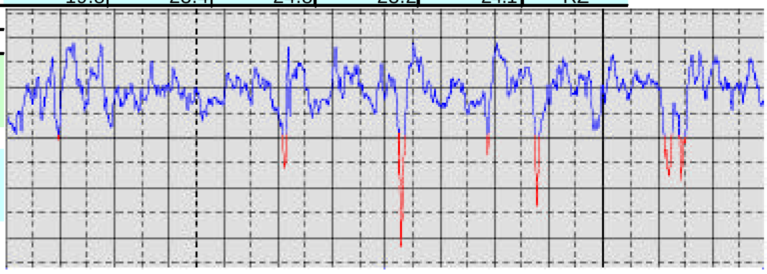
Ra	=	the arithmetic mean distance between the roughest peaks and valleys
Rz	=	the maximum height between the highest peak and deepest valley

Figure 9: Typical milled surface roughness profile using 10 µm spherical probe

		Messablauf 1	2	3	4	5	
Test piece	Nr.1						
Distance in mm	5.6mm	1.442	1.442	2.168	1.144	1.144	RA
Inspection spherical tool	10µm	6.332	6.332	8.604	6.382	6.382	RZ
Machining	Milling tool						
Status of tool	New						
RA MIN	1.144						
RA MAX	2.168						
RA Mitt	1.468						
RZ MIN	6.332						
RZ MAX	8.604						
RZ Mitt	6.8064						



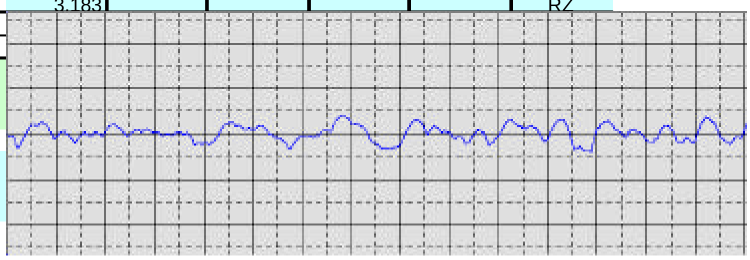
		Messablauf 1	2	3	4	5	
Test piece	Nr.2						
Distance in mm	5.6mm	3.12	3.55	3.46	3.52	3.2	RA
Inspection spherical tool	10µm	19.8	23.4	24.8	23.2	24.1	RZ
Machining	Milling tool						
Status of tool	Verschlissen						
RA MIN	3.12						
RA MAX	3.55						
RA Mitt	3.37						
RZ MIN	19.8						
RZ MAX	24.8						
RZ Mitt	23.06						



RA Arithmetical average of profile in vertical direction
RZ Max height of profile - considering the max. and lowest point of profile

Figure 10: Typical milled surface roughness profile using 1.5 mm spherical probe

		1	2	3	4	5	
Test piece	Nr.1						
Distance in mm	5.6	0.681					RA
Inspection spherical tool	1.5mm	3.183					RZ
Machining	Milling tool						
Status of tool	New						
RA MIN	0.681						
RA MAX	0.681						
RA Mitt	0.681						
RZ MIN	3.183						
RZ MAX	3.183						
RZ Mitt	3.183						



		Messablauf 1	2	3	4	5	
Test piece	Nr.2						
Distance in mm	5.6	1.027					RA
Inspection spherical tool	1.5mm	5.735					RZ
Machining	Milling tool						
Status of tool	Verschlissen						
RA MIN	1.027						
RA MAX	1.027						
RA Mitt	1.027						
RZ MIN	5.735						
RZ MAX	5.735						
RZ Mitt	5.735						

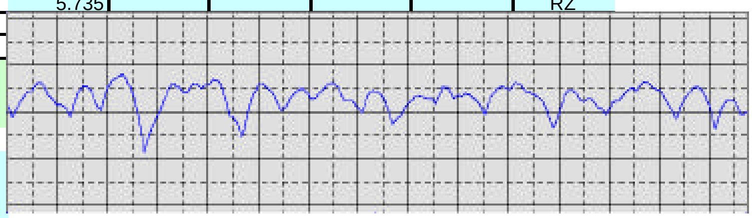
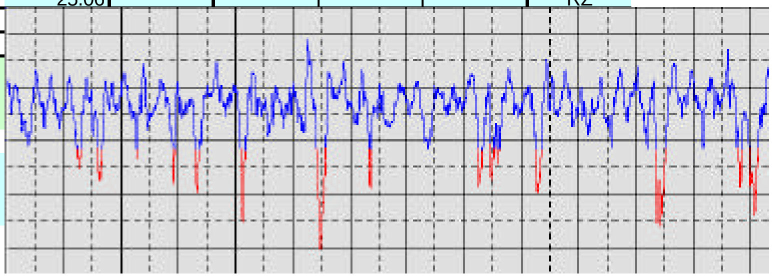


Figure 11: Typical turned surface roughness profile using 10 µm and 1.5 mm spherical probe

Distance of measuring

		1	2	3	4	5	
Test piece	Zugprobe						
Distance in mm	17.5	3.97					RA
Inspection spherical tool	10µm	25.06					RZ
Machining	Lathe						
Status of tool	New						
RA MIN	3.97						
RA MAX	3.97						
RA AVE	3.97						
RZ MIN	25.06						
RZ MAX	25.06						
RZ AVE	25.06						



Messablauf

		1	2	3	4	5	
Test piece	Zugprobe						
Distance in mm	17.5	2.769					RA
Inspection spherical tool	1.5mm	15.045					RZ
Machining	Lathe						
Status of tool	New						
RA MIN	2.769						
RA MAX	2.769						
RA AVE	2.769						
RZ MIN	15.045						
RZ MAX	15.045						
RZ AVE	15.045