

**NNC Contribution to HTR-E Project deliverable
D40: Proposal for elementary tribology tests**

By

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Table of Contents

List of Tables.....	iii
Summary.....	iv
1 Introduction.....	1
2 UK experience	1
2.1 Introduction.....	1
2.2 Friction and wear testing	1
2.2.3 Effect of dwells at elevated temperature.....	3
2.3 Static adhesion	3
2.4 Fretting	4
3 Tribological concerns in a HTR.....	5
4 Testing required.....	5
4.1 Introduction.....	5
4.2 Test recommendations	5
5 References	7

List of Tables

Table 1	Results from tests carried out during the Dragon project to investigate the effects of contact pressure, helium pressure and helium purity on friction and wear
Table 2	Friction and wear tests in helium with varying moisture content (Dragon Project)
Table 3	Fretting test experimental parameters (Mk 3 programme)
Table 4	Areas of tribological concern in a HTR of pebble bed design

Summary

This document reports on UK experience in support of elementary tribological tests to be carried out in support of the HTR programme. The report contributes to deliverable D40 of the HTR-E Project. Information relevant to test conditions and detailed recommendations are made for the conditions to be applied during the elementary tribological tests.

1 Introduction

Drawing on the experience gained in the UK during the test programmes for, and operation of, the Dragon, Magnox, AGR and PFR reactor systems, recommendations are made for elementary tribological tests to be carried out in support of the HTR programme. The report contributes to deliverable D40 of the HTR-E Project.

Tribological problems identified by UK studies were reviewed in D37 [1], and materials and coatings were reviewed in D39 [2]. In D38 [3], tribological concerns were identified for the Pebble Bed Modular Reactor. This report contains a synthesis of those documents, includes new information relevant to test conditions and makes detailed recommendations for the conditions to be applied during elementary tribological tests.

This document contains the results of work at NNC on Deliverable D40 of the HTR-E Project.

2 UK experience

2.1 Introduction

UK experience in helium tribology stems largely from the Dragon Project and tests programmes in support of a Mk III gas-cooled reactor. Extensive 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. In this section, there is an emphasis on helium tribology. The effects of different parameters are assessed and conclusions of relevance to an HTR test programme are drawn.

2.2 Friction and wear testing

2.2.1 Effects of gas pressure, contact pressure and helium purity

During the Dragon Project, friction and wear testing was carried out in a reciprocating sliding configuration in 'pure' and 'impure' helium at temperatures of up to 800°C [4]. Tests in pure helium were carried out 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.

In carrying out a test, the circuit would be evacuated and then filled with helium, which was circulated and purified as it passed through a liquid nitrogen-cooled molecular sieve. With full flow through the purifier, circulation for two hours was sufficient to reduce the levels of water, oxygen and nitrogen to 2, 0.2 and 1 vpm, respectively. After the initial purification, a large proportion of the gas was allowed to bypass the purifier to conserve liquid nitrogen. The water level was measured using an electrolytic hygrometer while the oxygen and nitrogen levels were determined using a gas chromatograph.

For tests in 'impure' helium, a gas plant was used to supply helium with impurity levels of 25 vpm hydrogen, 25 vpm CO and 2.5 vpm water. Apparatus limitations restricted the test pressure to 20 bar when using 'impure' helium.

The following test conditions were employed:

- Specimen size stationary: 12.7 mm diameter, 6.35 mm thick

reciprocating: 25.4 mm diameter, 6.35 mm thick

- Apparent contact pressure 0.7 – 6.5 MPa
- Stroke 10 mm
- Cycling rate 1.5 Hz
- Number of cycles 5000
- Total rubbing distance 100 m
- Duration of test 54 minutes
- Gas circulation rate 2.7 litres/minute

Many materials combinations were tested, results from which were presented previously [1] [2]. However, five materials combinations were chosen specifically to investigate the effect of helium pressure and purity on friction and wear. These combinations were considered typical of many rubbing pairs in Dragon.

1. Type 316 stainless steel, like-on-like
2. D-Gun coating LC1B, like-on-like
3. Gilsocarbon, like-on-like
4. Nitrided En 40B, like-on-like
5. Alloy C weld deposit vs Type 316 stainless steel

A summary of the experimental results is presented in Table 1. It was concluded from the programme of friction and wear tests that there was no significant difference between wear behaviour in pure and impure helium. In tests in pure helium, there was no systematic effect of gas pressure on wear rate. The highest friction coefficients were measured in pure dry helium. The concentration of water and oxygen in the impure helium had a beneficial influence on the coefficient of friction. This was attributed to the formation of lubricating oxide films.

During the AGR programme, friction and wear data were gathered from reciprocating sliding tests [5]. The reciprocating sliding tests were normally conducted on specimens in flat-on-flat configuration, with apparent contact pressures in the range 1 – 10 MPa. A stroke of 12.7 mm at a frequency of 1 Hz was employed. Testing was carried out at gas pressures of 2 – 42 bar. A crossed-cylinder arrangement was used for some tests on thick coatings, e.g. weld overlays.

The specific wear rates of LC1B and LW5 in CO₂ decreased with duration of rubbing. The wear rates were highest at temperatures of 200 – 450°C and lowest at 500 – 650°C, and were dependent on gas pressure. The lowest wear rates occurred in tests at the highest pressures.

2.2.2 Effect of moisture

For the Dragon Project, some tests with varying moisture content were carried out at 20 bar gas pressure on 316 stainless steel, like-on-like and vs Alloy C: the results are presented in Table 2 [4]. At both test temperatures (515°C and 650°C) the friction coefficients reduced progressively with increasing moisture content. There was no systematic effect on wear.

The moisture content of the gas was found to influence the running of plain bearings used in Dragon [6]. Most of these operated at light loads and slow speeds, at operating temperatures between 350°C and 500°C. In bearings tests on metal, ceramic, plastic, carbon and graphite bearings, the water content had an important influence on running. When the vapour concentration was greater than 100 vpm most materials ran together without seizure. When the water vapour concentration was reduced to below 20 vpm, all materials tested at temperatures of 350°C and above either seized immediately (metals and ceramics) or wore excessively (graphite and carbons). The only way to reduce the wear rate was by using molybdenum disulphide coatings. The only satisfactory plain bearing combination found for operation at elevated temperature in pure helium was molybdenum disulphide-treated carbon or graphite operating in conjunction with a well-finished hard material.

2.2.3 Effect of dwells at elevated temperature

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. In order to assess the effects of dwells at temperature in helium, a series of reciprocating sliding tests was performed at temperatures between 450°C and 750°C [7]. Each materials combination was tested in 'impure' helium at a gas pressure of 1.68 bar. Each materials combination was rubbed continuously for 100 cycles at 0.5 Hz and the results were compared with those obtained from dwell tests where the materials were run for 5 cycles each day for 20 days. Several alloys and one coating (LC1B) were tested.

Alloy C/316, 316/316, Nimonic 90/316 and Nimonic 90/Nimonic 90 all showed little change of dynamic friction due to dwell. However, the initial values of static friction measured after dwell periods could be very high. Wear, mostly in the form of material transfer, tended to be greatest in the dwell tests. LC1B, like-on-like gave the best overall performance in these tests.

2.3 Static adhesion

Small rectangular specimens of a large range of materials were subjected to dead weight loading for extended periods in an autoclave [8] [9]. Tests were carried out in 'pure' and 'impure' helium at temperatures between 400°C and 800°C for periods of 200 h and 1000 h. A pressure of 0.3 MPa was applied to the specimens. Following testing, any adhering specimens were parted in a tensometer. Selected specimens were subjected to detailed metallography. Materials included stainless steels, mild and low alloy steels, Nimonic alloys, Incoloy 800, hard facing alloys, flame sprayed alloys, graphites and alumina.

At 400°C, the specimens showed little or no tendency to adhere. Adhesion was found to increase with temperature and time at temperature. At temperatures between 650°C and 800°C all metallic pairs except Nimonic 90 vs En 58J austenitic stainless steel exhibited static

adhesion. The greatest degree of static adhesion was found in couples of En 58J stainless steel. After 1000 h exposure at 800°C, a parting pressure of 7 MPa was required. The presence of impurities in the gas decreased the adhesion below that found in pure helium. Alumina and graphite showed little tendency to bond, either to themselves or to metallic specimens. Detonation-Gun coated LC1B adheres both to itself and to metal surfaces at 800°C; the degree of adhesion depends on the surface roughness.

2.4 Fretting

Impact fretting tests were performed at temperatures from 20°C to and 750°C in helium at 1.24 bar gas pressure [10]. A hammer specimen was attached to a vertically vibrating shaft so that it impacted against two angled anvil specimens which gave way on impact to allow a sliding component between the hammer and anvil. The contact between hammer and anvils was flat-on-flat. The experimental parameters are presented in Table 3. To assess the effects of helium purity, tests were carried out in 'pure' helium (<0.1 Pa H₂O) and in 'impure' helium (5 Pa H₂O, 5 Pa CH₄, 50 Pa CO and 50 Pa H₂), representative of a boiler leak condition.

The following materials combinations were tested:

1. Type 316 stainless steel, like-on-like
2. 316 stainless steel vs Alloy C
3. Incoloy 800 vs 316 stainless steel
4. 316 stainless steel vs 2¼ Cr 1 Mo
5. Gilsocarbon, like-on-like

In addition, commissioning tests were carried out on En 58B austenitic stainless steel at 75 Hz, like-on-like, from which the following conclusions were drawn:

- A 5 hour test (1.35×10^6 cycles) in helium at 600°C was not sufficient to produce measurable wear.
- The tests require a running-in period, during which the wear rate is very low, of up to 20 h.

Conclusions from the scheduled tests are presented below:

- With the possible exception of 316 stainless steel, like-on-like, wear in impure helium was not significantly different from that observed in pure gas. However, graphite was not tested in pure gas.
- The heaviest wear for all combinations occurred at room temperature. The least wear occurred over the temperature range 350 – 750°C. Incoloy 800 vs 316 stainless steel showed the smallest variation.
- No clear evidence was found of a frequency effect on wear rate.
- Long term ($\sim 10^3$ h) fretting tests are essential.

3 Tribological concerns in a HTR

Many components in both Dragon and the AGR were identified as being susceptible to wear due to sliding contact and fretting [1] [2]. Sliding contacts arise during the insertion and withdrawal of components within the reactor and as a consequence of expansion and contraction upon thermal and load cycling. During periods in which no relative motion occurs, especially at small area contacts, static adhesion may follow: fatigue or component seizure may then result. The galling of fasteners during protracted exposure to an inert atmosphere at elevated temperature was another area identified for concern. Fretting may arise between contacting parts in high velocity turbulent gas flow. In the heat exchangers, impact/slide fretting was a major concern.

A review of typical HTR tribological conditions, focusing on the PBMR type reactor design, was made previously [3]. Specific areas of concern are presented here in Table 4 together with recommendations for the testing required.

4 Testing required

4.1 Introduction

Building on the UK's experience of tribological issues in helium atmospheres, and in consideration of the tribological concerns in a future HTR, recommendations have been made for preliminary tests. More comprehensive testing will ultimately be required, including tests for specific components, e.g. bearings.

4.2 Test recommendations

Sliding, fretting and static adhesion tests will be required. To cover the range of potential impurity levels, tests will be required in gases of different composition, in particular different moisture content. Different test pressures will also be required; for preliminary tests: the highest practicable pressure should be employed. Temperature dwells should be incorporated into tests involving parts in relative motion.

Recommended materials and test conditions are set out below. The values of test parameters are guidelines only.

4.2.1 Materials and coatings

Materials: Component alloys, e.g. nickel-based alloys and stainless steels

Graphite (vs graphite; vs steel)

Hard coatings:

D-GunWC/Ni/Mixed W/Cr carbides
(LW5 type); $T \leq 350^{\circ}\text{C}$

D-Gun $\text{Cr}_3\text{C}_2/\text{Ni-Cr}$ (LC1B type); $T \leq 700^{\circ}\text{C}$

Plasma sprayed $\text{Cr}_3\text{C}_2/\text{Ni-Cr}$ (LC2 type);
 $T \leq 700^{\circ}\text{C}$

$\text{Cr}_{23}\text{C}_6\text{-Cr}_{23}\text{C}_6/\text{Ni-Cr duplex}$; $T \leq 850^\circ\text{C}$

Plasma sprayed Y stabilised ZrO_2 on NiCrAlY ;
 $T \leq 950^\circ\text{C}$

Lubricating coatings: MoS_2 ; different formulations to be assessed at all temperatures in friction and wear tests

Graphite; to be assessed at all temperatures in friction and wear tests

4.2.2 Gas parameters

Composition: (a) Pure; water content ≈ 0.1 vpm; other impurities: CO (2 vpm) and H_2 (20 vpm)

(b) Impure; water content ≈ 2 vpm;
other impurities: CH_4 (2 vpm), CO (20 vpm) and H_2 (20 vpm); continuous gas monitoring recommended

Pressure: ≈ 70 bar or, if lower, as high as practicable

Flow: Circulating gas, continuously purified if feasible

The effect of small quantities of oxygen on the tribology will need to be considered at some stage.

4.2.3 Friction and wear tests

Movement: Reciprocating sliding (pin-on-plate); stroke ≈ 10 mm; frequency ≈ 1 Hz; $10^3 - 10^4$ cycles; incorporate dwells at temperature in some tests

Continuous sliding (pin-on-disc); sliding distance $\approx 10^3$ m

Contact pressure (load/pin area): $\approx 0.2 - 1$ MPa; $\approx 5 - 10$ MPa

Specimen roughness: $R_a \approx 0.4$ μm or as appropriate

Temperature: $20^\circ\text{C} - 800^\circ\text{C}$ or as high as achievable

Measurements: Dynamic friction coefficient; static friction coefficient during standard tests and after dwells; weight change; wear depth

Some consideration should be given to the pre-exposure of coatings to reactor gas.

4.2.4 Static adhesion

Contact pressure (load/area):	$\approx 0.2 - 0.5$ MPa, initially
Specimen roughness:	$R_a \approx 0.2$ μm or as appropriate
Temperature:	400°C – 900°C
Duration:	$\approx 10^2 - 10^3$ hours
Measurements:	Parting load

In addition to simple static adhesion tests, a programme of fastener torque-tension testing after exposure to high pressure helium will be required at some stage.

4.2.5 Fretting

Movement:	Sliding; stroke ≈ 0.2 mm; frequency $\approx 50 - 150$ Hz; $\approx 10^7 - 10^9$ cycles; impact-slide if available
Contact pressure (load/pin area):	$\approx 0.1 - 0.5$ MPa
Specimen roughness:	$R_a \approx 0.4$ μm or as appropriate
Temperature:	20°C – 800°C or as high as achievable
Measurements:	Weight loss; wear depth; metallography of cross-sections

5 References

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Table 1 Results from tests carried out during the Dragon project to investigate the effects of contact pressure, helium pressure and helium purity on friction and wear

Materials	Temp (°C)	Friction coefficient								Specific wear rate ($\times 10^{-14} \text{ m}^3/\text{Nm}$)						
		Contact pressure (MPa); tests in pure helium at 1 bar				Pure He gas pressure (bar)			Impure He gas pressure (bar)	Contact pressure (MPa); tests in pure helium at 1 bar				Pure He gas pressure (bar)		Impure He gas pressure (bar)
		0.7	2.1	3.8	6.5	1	20	50	20	0.7	2.1	3.8	6.5	1	50	20
316, like-on-like	650	4	3	2		2.5	2	2.5	2							
	800		2					3	8							
LC1B, like-on-like	20		0.6		0.7						30		19			
	800		0.7		1.0	1.0		0.6	0.7		<1		<1	<1	<1	<1
Gilsocarbon, like-on-like	20		0.2		0.3	0.3		0.3	0.3		1		60	60	60	50
	400		0.3		0.2	0.2		0.2	0.1		4		2	2	10	2
	800		0.3		0.4	0.3		0.4	0.4		5		2	1	1	3
Nitrided En 40B, like-on-like	20				0.4/0.7											
	400		0.9		0.6/1.0	0.6/1.0		0.6/1.0	0.6/0.8				15	15	3	<1
Alloy C vs 316	20				1			1.4					5	5	5	
	650		2		2		3	2	2							
	800				5				2.5							

Table 2 Friction and wear tests in helium with varying moisture content (Dragon Project)

Materials	Impurity content (vpm)			Temp (°C)	Friction coefficient			Specific wear rate ($\times 10^{-14}$ m ³ /Nm)
	H ₂ O	H ₂	O ₂		Minimum	Maximum	Typical	
316 stainless steel, like-on-like	2.0	2.3	0.1	650	1.67	2.47	2.1	24
				515	1.48	2.67	2.0	82
	25	270	0.6	650	1.52	2.20	1.8	14
				515	1.36	2.02	1.65	60
	65	480	12	650	1.08	1.56	1.4	43
				515	1.09	1.52	1.25	22
316 stainless steel vs Alloy C	1.7	4	0.6	650	2.26	3.1	2.8	34
				515	1.82	3.3	2.8	29
	26	260	0.5	650	1.63	2.68	2.4	24
				515	1.80	2.70	2.2	36
	48	430	0.7	650	2.04	2.46	2.2	19
				515	1.68	2.29	1.9	48

Table 3 Fretting test experimental parameters (Mk 3 programme)

Test parameter	Frequency		
	75 Hz	150 Hz	
Test duration (h)	30	30	50
Total number of cycles	8.1×10^6	1.6×10^7	2.7×10^7
Peak normal impact sliding load (N)	50	25	25
Vibration amplitude, peak-to-peak (mm)	0.254	0.152	0.152
Slip amplitude, peak-to-peak (mm)	0.180	0.107	0.107
Total sliding distance (m)	2910	3480	5800
Total contact time (h)	15	24	40

Table 4 Areas of tribological concern in a HTR of pebble bed design

Component	Area	Temperature (°C)	Motion	Testing required
Heat exchangers	Pre-cooler. Intercooler.	<150	Thermal movement. Vibration.	Fretting. Impact/slide fretting. Reciprocating sliding.
Turbo units	Bearings	700	Sliding	Fretting. Continuous sliding.
	Blades			Erosion
Reactivity control system	Control rod		Sliding	Continuous sliding. Reciprocating sliding. Static adhesion.
Core	Pebbles, blocks, bricks, keys, supports, steel barrel	450-900	Thermal movement. Sliding.	Reciprocating sliding. Fretting.
Various	Valves	Various	Sliding	Static adhesion. Reciprocating sliding with temperature dwells.
Various	Seals	Various	Thermal movement. Sliding.	Reciprocating sliding.