Lars Ekerfors

Tribological Testing of Graphite in a Wet Environment

SP
Swedish National Testing and Research Institute
Mechanics
SP REPORT 1997:01

Abstract

A tribological method of testing was developed to rank a number of similar materials with respect to wear in a wet environment. This was achieved by applying gradual experimental planning.

Tests were performed in a Pin-on-Disc machine modified for a wet tempered environment. The tribo-couple was graphite versus steel immersed in water. The contact was sliding and exposed to a normal load.

The significant parameters in the present tribo-system are contact pressure, sliding velocity, temperature, the surface roughness of the thread flanks, the type and mass of particles of wear and, finally, the quality of graphite.

Initially the effect of the parameters was unknown, which called for gradual experimental planning.

Key words: tribology, experimental planning, graphite, wear

Sveriges Provnings- och Forskningsinstitut
SP Rapport 1997:01
ISBN 91-7848-659-9
ISSN 0284-5172
Borås 1997

Swedish National Testing and Research Institute
SP Report 1997:01

Postal address:
Box 857, SE-501 15 BORÅS
Sweden
Telephone +46 33 16 50 00
Telex 36252 Testing S
Telefax +46 33 10 20 15

Contents

Abstract 2

Contents 3

1. Introduction 5

2. Test equipment 6

3. Data and data processing 9

4. Test procedure 10

5. Stage A 11
5.1 Testing parameters 11
5.2 Results 12
5.3 Summary of results 15

6. Stage B 16
6.1 Tested qualities of graphite 16
6.2 Results 16

7. Conclusions 24

8. Acknowledgement 25

1. Introduction

In cooperation with ABB Atom AB, the Swedish National Testing and Research Institute has performed comparative tribological testing of the wear of different qualities of graphite. The objective of the testing was to rank 14 qualities of graphite regarding the wear in a wet environment. The tribo-couple was graphite versus steel immersed in water. The contact was sliding and exposed to a normal load.

The tests were performed with a pin-on-disc machine where the tribological conditions in the driving devices of the control rods in a nuclear reactor were simulated as far as possible. The driving device consists of a screw/nut transmission working in pressurized water of different temperatures. In certain reactors, the temperature of the flushing water can be 240 °C. In the transmission, the flanks of the threads of the steel screw and graphite nut slide on each other when carrying the axial load from the screw itself and the control rod. Furthermore, the tribological environment is characterized by crystals of oxide appearing on the surfaces of the flanks of the screws and also by particles in the flushing water. Due to this fact the thread flanks of the steel screw have varying surface roughness.

In view of these facts the significant parameters in the present tribo-system are contact pressure, sliding velocity, temperature, the surface roughness of the thread flanks, the type and mass of particles of wear and, finally, the quality of graphite.

The testing was carried out in two stages, stage A and stage B.

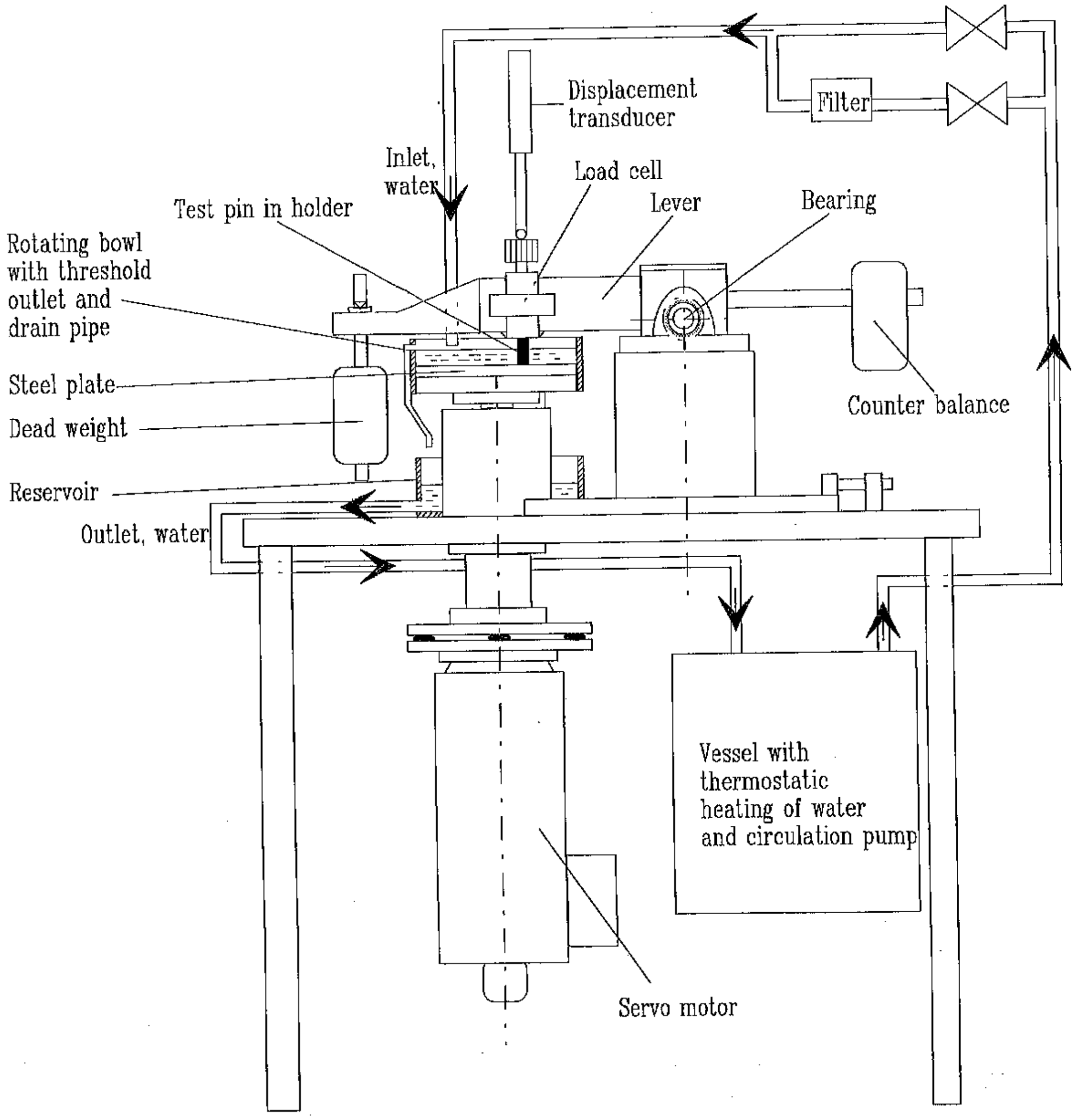
The objective in stage A was to make pretests to establish the most representative values and combinations of the parameters mentioned above with reference to wear. This was done by applying gradual experimental planning. Here one acts on combinations of values, which exist within a range typical for the real system. The combinations are gradually and systematically chosen, guided by the analysis of the results from the continuous testing. The varied parameters were contact pressure, surface roughness and particles in the process water. The varied surface roughness was accomplished by testing on surfaces of steel prepared in different ways. A specification of the applied parameters is presented in chapter 5.1 Testing parameters.

In stage B comparative wear experiments with 14 separate qualities of graphite were carried out with the established parameters.

2. Test equipment

The tests were performed with a VTT Pin-on-Disc-machine, 100 N, modified for a wet, tempered environment. With this machine, the wear of the test specimen is measured when it slides on a rotating disc of steel. The test specimen consists of a pin of graphite. The wear is defined as the worn volume of graphite when the pin declines and can be measured as the reduction of length in mm. In addition to wear, the frictional force between pin and disc can be measured. Figure 1 shows the fundamental construction of the test equipment.

Figure 1. Diagram of the Pin-on-Disc-machine for testing in a wet and tempered environment



Data were recorded and processed with the following equipment:

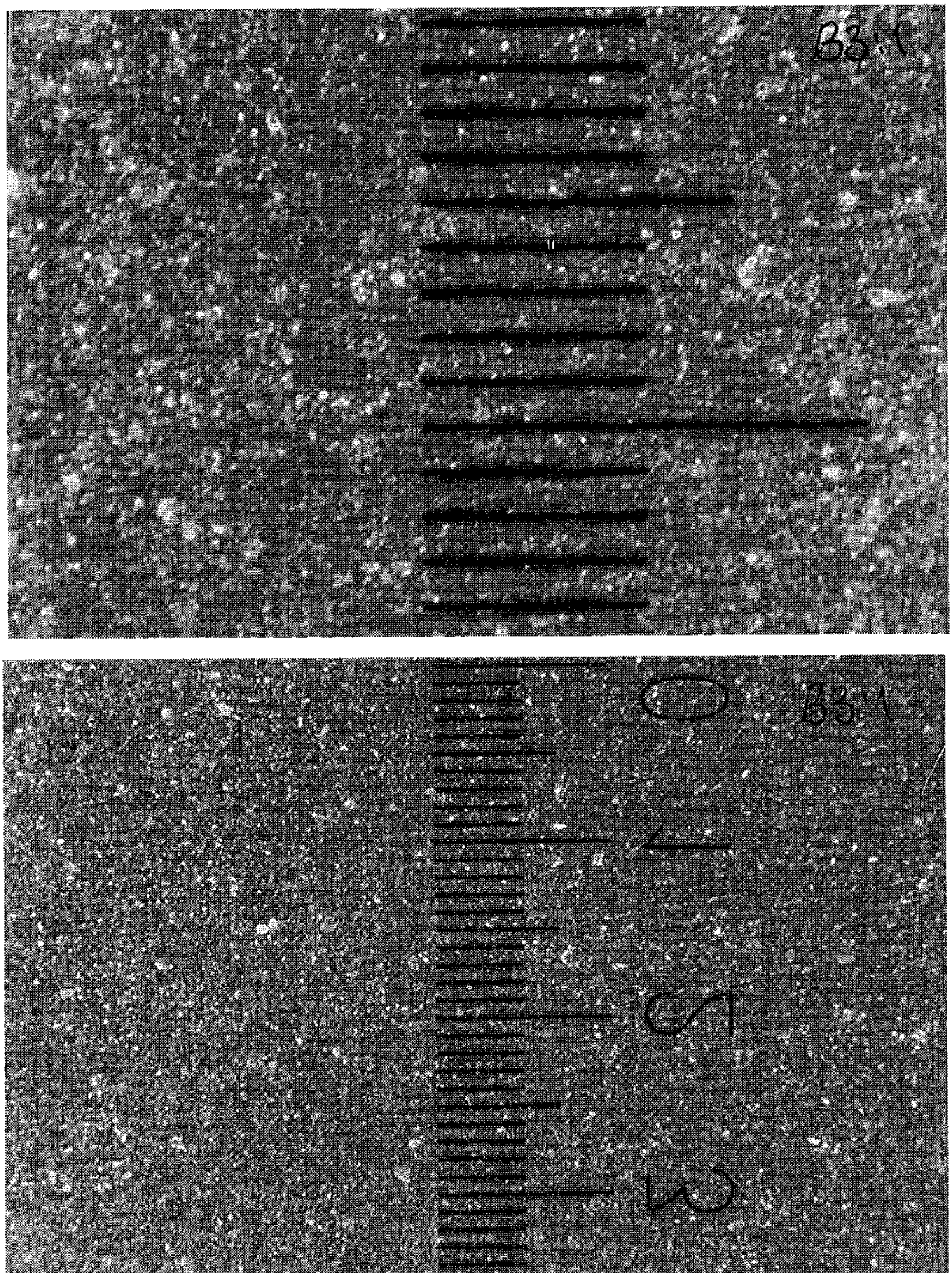
load cell	HBM Z6C3 cap 100 N
amplifier	Nobel B KI 5
displacement transducer	HBM W10 TK ± 10 mm
signal conditioner	HBM MC 2
data acquisition board	Compaq 4 86 MHz
data acquisition program	Dasy-Lab 2.0
surface roughness tester	Mitutoyo SURFTEST 301

To establish possible remaining abrasive agents or possible products of wear on the end surface of the graphite pin and the steel disc, a 3D-microscope, Wild M8 No. 400500 was used. No remnants of abrasives were revealed in any of the graphite qualities tested. Enlargement of the end surface of one of the graphite pins is shown in figure 2.

On the other hand, ocular inspection showed that wear products in the form of graphite powder stuck to the steel discs to a varying extent while the testing was in progress.

In the tribo-couple in question the following steel qualities were applied: SS 2321 (tempered) and SS 2333. Two alternative surface roughnesses were applied: smooth (ground) surface and blasted surface.

Figure 2. Enlargement (×18 and ×50) of the end surface of graphite pin, quality $(Gr\ 2)_{R,}$ smooth disc + addition of AlO (13 -20 μm), contact pressure 0.25 N/mm^2, sliding distance 1.44 km.



3. Data and data processing

The significant recorded quantity is the wear of the graphite pin. The wear is defined as the reduction of length in mm which can be measured/recorded during testing. The wear was measured in two different ways, partly by recording the lowering of the pin with a displacement transducer, partly by measuring the length of the pin before and after testing with a digital slide-calliper, i.e. the total wear during the time of testing.

The friction force was recorded with a load cell, and this was done at the same time as the recording with the displacement transducer.

The signals from the displacement transducer and the load cell were sampled at 100 Hz and scaled after the signal conditioner/amplifier. The mean of sampled data was calculated in blocks of 250 values. Accordingly, such a calculated value corresponds to the mean of 250 sampled values. These means are sampled in a data file and plotted via the computer program Excel 5.0 in diagrams. In these diagrams, the wear and the friction force are plotted versus testing time.

Significant of the wear process is that, after a certain running-in period, the wear reaches steady state, i. e. a constant rate of wear. This is seen in the wear diagram as a straight line with a constant inclination. The inclination was used in the presentation of the result to calculate the specific wear, which is defined as wear per sliding distance, mm/km, i. e. a dimensionless number typical of the quality of graphite in question.

The length of the running-in period, which in the wear diagram corresponds to the distance from the starting-point to the intersection between the running-in curve and the steady state line, varies for the different qualities of graphite.

For some qualities steady state was never reached during the total sliding distance in these tests, i.e. the intersection is beyond the wear diagram.

The calculation of the specific wear in this report is based upon the latter half of the wear curve, which corresponds to the wear process during the sliding distance from 720 m to 1440 m.

It can be seen from the results that, for most of the qualities, the steady state line with a wide margin has been reached. For those qualities where the intersection lies beyond the wear diagrams, inspection of the curves in the results shows that the calculation is a good approximation.

4. Test procedure

The following test procedure was applied to both stages A and B. The preparation for each test comprises:

Heating of the process water to 70 °C.
The length of the graphite pin is measured and recorded.
The graphite pin is fit in its holder.
The disc is mounted. This allows checking that the disc is perpendicular to the axis of rotation.
When testing with smooth surface, the abrasive agent is mixed with the process water to a concentration of 25 g per 4 l.
The displacement transducer is mounted.
The pin is loaded.
The disc is started and the rotational speed is set.
The electronic equipment is switched on.

The test includes:

Regular surveillance.
Obliqueness of the graphite pin is continuously adjusted.
The electronic equipment is switched on.

The test is interrupted after 2 h, which corresponds to 1.44 km sliding distance.
The length of the graphite pin is measured and recorded.
The disc is dismounted and cleaned.

Before and after the test, the surface roughness (Ra) of the sliding surface of the steel disc is measured.

5. Stage A

5.1 Testing parameters

Two qualities of graphite of different hardness were chosen for the present stage. These qualities serve as references and are here called Gr 1 and Gr 2.

The totally desalted process water was delivered by ABB.

The applied testing parameters are specified below:

Contact pressure:	0.1 0.2 0.25 0.4 and 0.5 N/mm^2
Sliding velocity:	0.2 m/s
Temperature:	70°C
Surface conditions:	Smooth disc Ra 0.25 Smooth disc + addition of AlO (13 - 20 μm) to the process water Smooth disc + addition of SiC (4 - 5 μm) to the process water Wet grinding paper (granularity 360) Blasted disc (manual blasting with SiC 30) Blasted disc + addition of AlO (13 - 20 μm) to the process water (manual blasting with SiC 30) Blasted disc + addition of SiC (4 - 5 μm) to the process water (manual blasting with SiC 30)
Graphite qualities:	*Gr 1 and Gr 2*
Quality of steel:	SS 2333
Test periods:	0.5 - - 6 h

To establish adequate parameters of blasting, blasted discs were tested according to the following:

Pressure of blasting:	4, 6 and 8 bar
Velocity of blasting:	manual blasting mechanically controlled blasting, 0.12 m/s
Blasting agents:	SiC 30 SiC 46 SiC 60 AlO 46
Quality of steel:	SS 2321 (hardened)

5.2 Results

The parameters in question were tested in the combinations shown in the tables below. The results, presented as the total wear, in mm, from the testing periods, are also given.

Compilation of pretests, tables 1 - 4, in all 52 tests.

Table 1. Tests with different contact pressure, type of disc and time. No addition of abrasive agents.

Contact pressure N/mm^2	Surface of disc	Pin quality	Testing period	Wear mm
0.1	smooth	Gr 1	60 min	**0.0**
	Slip 360	Gr 1	30 min	**< 1.0**
	Blasted	Gr 1	(60 min)	**(2.6)**
			6 h	**3.8**
		Gr 2	5.5 h	**0.0**
0.2	Blasted	Gr 1	2h	**8.1**
		Gr 1	(5 min)	**(1.4)**
			60min	**13.9**
		Gr 2	60 min	**< 0.1**
		Gr 1	70 min	**14.2**
		Gr 2	60 min	**0.1**
	Slip 360	Gr 1	40 min	**1.6**
		Gr 2	60 min	**1.0**
0.4	Smooth	Gr 1	60 min	**0.0**
	Slip 360	Gr 1	60 min	**2.5**
		Gr 2	60 min	**1.4**
	Blasted	Gr 1	35 min	**16.9**
		Gr 2	60 min	**0.1**

Table 2. Tests with different concentration of the abrasive agent AlO.

Contact pressure N/mm^2	Pin quality	Testing period min	Surface of disc + abrasive agent (total quantity per 4 l water)	Wear mm
0.25	Gr 2	120	Blasted +10 g AlO	**23.1**
			Blasted + 50 g AlO	**31.0**
		58	Smooth + 50 g AlO	**2.7**

Table 3. Tests with different abrasive agents, contact pressure and type of disc.

Test period	Abrasive agent		Contact pressure	Surface of disc	Pin	Test	Wear
min	total quantity g	type	N/mm^2		quality	no.	mm
60	25	AlO	0,25	Blasted	Gr 2	1:1	**20.1**
						1:2	**19.0**
					Gr 1	2:1	**18.0**
				Smooth	Gr 2	3:1	**3.7**
					Gr 1	4:1	**0.8**
						4:2	**0.9**
			0.5	Blasted	Gr 2	5:1	**19.5**
					Gr 1	6:1	**19.1**
				Smooth	Gr 2	7:1	**4.4**
						7:2	**5.3**
					Gr 1	8:2	**0.7**
						8:3	**0.6**
						8:4	**0.5**
		SiC 1200	0.25	Blasted	Gr 2	9:1	**6.0**
					Gr 1	10:1	**3.6**
				Smooth	Gr 2	11:1	**1.9**
						11:2	**1.9**
					Gr 1	12:1	**0.5**
			0.5	Blasted	Gr 2	13:1	**5.5**
					Gr 1	14:1	**4.9**
						14:2	**4.4**
				Smooth	Gr 2	15:1	**1.7**
					Gr 1	16:1	**0.3**

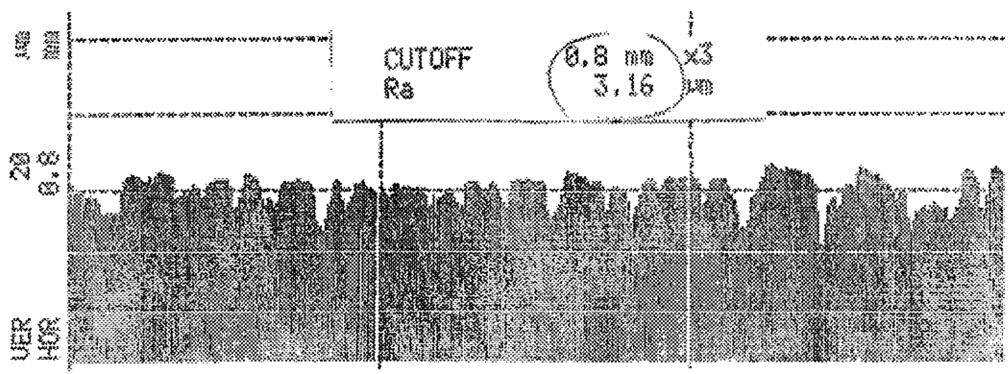
Table 4. Tests with different abrasive agent and test periods.

Contact pressure	Surface of disc	Abrasive agent		Test period	Pin	Test	Wear
N/mm^2		quantity	quality	min	quality	no.	mm
0.25	smooth	25 g	SiC grain 180	60	Gr 2	B1:1	**7.8**
						B1:2	**5.4**
					Gr 1	B2:1	**3.3**
						B2:2	**2.9**
			AlO	120	Gr 2	B3:1	**7.3**
						B3:2	**9.2**
					Gr 1	B4:1	**0.9**
						B4:2	**1.0**

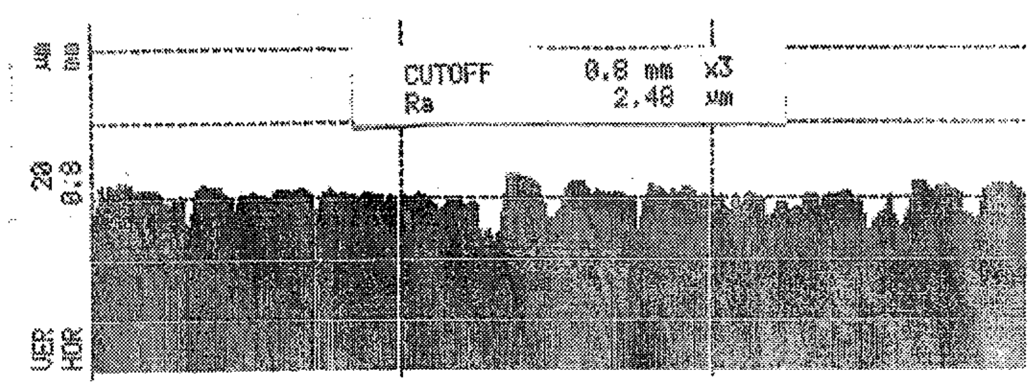
The diagrams of surface roughness from the pretests with different parameters of blasting are presented in figure 3.

Figure 3. Diagrams of surface roughness from mechanically controlled blasting, velocity 0.12 m/s, and blasting agent SiC 46. Pressures of blasting: 8, 6 and 4 bar.

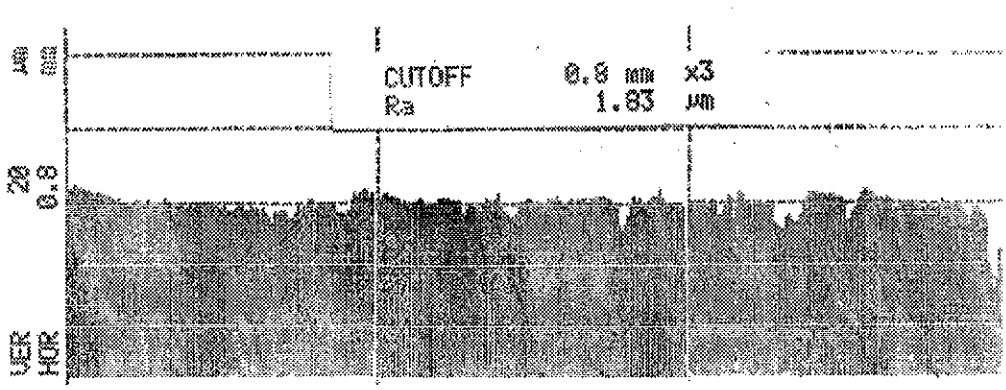
8 bar



6 bar



4 bar



5.3 Summary of results

On the basis of the results from stage A, it was decided that the comparative testing in stage B should be carried out along two main lines concerning the surface conditions of the steel discs:

•B:1 Smooth, ground surface, Ra 0.25 ± 0.05 μm + addition of AlO (13 - 20 μm) in the process water. Hardened steel, SS 23 21.

•B:2 Blasted surface, Ra 3.25 ± 0.50 μm. Hardened steel, SS 23 21.
Parameters of blasting: velocity 0.12 m/s
pressure 8 bar
blasting agent SiC 46

Moreover, the following testing parameters were established for the continued testing in stage B.

Contact pressure:	0.25 N/mm^2
Sliding velocity:	0.2 m/s
Temperature:	70°C
Test period:	2 h.

With the chosen value of the sliding velocity, a test period of 2 hours corresponds to the sliding distance 1.44 km.

6. Stage B

6.1 Tested qualities of graphite

Applying the results from section 5.3, comparative testing of the following 14 qualities of graphite was performed: $(\text{Gr } 1)_R$, $(\text{Gr } 2)_R$, Gr 3 ---- Gr 14.
The qualities $(\text{Gr } 1)_R$ and $(\text{Gr } 2)_R$ are the reference qualities tested in stage A.

6.2 Results

The results from stages B:1 and B:2 are presented as specific wear in tables 5 and 6 and the column diagram in figure 4 below.

The wear histories for each one of the 14 qualities of graphite in stages B:1 and B:2 are shown in diagrams in figures 5 and 6. Each curve of wear represents the mean of three tests for each quality of graphite.

Table 5. Total and specific wear, stage B:1, smooth surface

Quality of graphite	Total wear sliding distance 1.44 km (mm)	Spec. wear slid. period t_{60}-t_{120} (mm/km)	Spec.wear, mean (mm/km)
$(Gr\ 2)_R$	6.32	4.68	**3.93**
	7.15	4.50	
	5.21	2.60	
$(Gr\ 1)_R$	1.49	1.05	**0.89**
	0.92	0.49	
	1.60	1.12	
Gr 3	0.95	0.70	**0.38**
	0.37	0.23	
	0.78	0.22	
Gr 4	1.75	1.50	**1.02**
	0.72	0.50	
	0.78	0.42	
	1.63	1.66	
Gr 5	0.54	0.36	**0.87**
	0.75	0.41	
	2.21	1.83	
Gr 6	1.40	1.00	**0.67**
	0.80	0.63	
	0.49	0.38	
Gr 7	2.90	1.99	**1.81**
	2.17	2.12	
	1.24	1.33	
Gr 8	0,34	0.22	**0.22**
	0.30	0.21	
	0.38	0.24	
Gr 9	6.77	4.62	**2.65**
	2.47	1.60	
	3.43	1.74	
Gr 10	1.09	0.71	**0.73**
	1.01	0.77	
	1.39	0.70	
Gr 11	4.01	2.94	**2.14**
	3.79	2.32	
	2.58	1.17	

Table 5. Total and specific wear, stage B:1, smooth surface, (cont.)

Quality of graphite	Total wear sliding distance 1.44 km (mm)	Spec. wear slid. period t_{60}-t_{120} (mm/km)	Spec. wear, mean value (mm/km)
Gr 12	1.38	1.31	
	2.44	1.89	**1.65**
	1.70	1.74	
Gr 13	0.74	0.62	
	1.98	1.50	**1.20**
	1.78	1.46	
Gr 14	0.99	0.81	
	0.39	0.30	**0.52**
	0.66	0.46	

Table 6. Total and specific wear, stage B:2, blasted surface

Quality of graphite	Total wear sliding distance 1.44 km [mm]	Spec. wear slid. period t_{60}-t_{120} [mm/km]	Spec. wear mean [mm/km]
$(Gr\ 2)_R$	0.06	0.01	
	0.05	<0.01	**0.01**
	0.05	0.01	
$(Gr\ 1)_R$	4.75	0.41	
	4.90	0.40	**0.47**
	4.63	0.61	
Gr 3	0.95	0.07	
	1.80	0.11	**0.09**
	1.54	0.11	
Gr 4	0.52	0.03	
	0.52	0.03	**0.03**
	0.50	0.03	
Gr 5	29.15	12.23	
	26.64	11.24	**11.48**
	27.4	10.96	
Gr 6	28.57	4.36	
	25.11	3.66	**3.12**
	17.23	1.34	
Gr 7	0.06	<0.01	
	0.10	<0.01	**<0.01**
	0.22	<0.01	
Gr 8	7.70	0.30	
	12.05	1.03	**0.68**
	11.66	0.71	
Gr 9	0.04	<0.01	
	0.05	0.01	**0.01**
	0.12	0.01	
Gr 10	32.48	10.51	
	21.47	4.00	**6.71**
	23.90	5.62	
Gr 11	0.19	0.01	
	0.16	0.01	**0.01**
	0.10	<0.01	

Table 6. Total and specific wear, stage B:2, blasted surface, (cont.)

Quality of graphite	Total wear sliding distance 1.44 km (mm)	Spec. wear slid. period t_{60}-t_{120} (mm/km)	Spec. wear, mean value (mm/km)
Gr 12	0.330	0.02	
	0.17	0.01	**0.01**
	0.16	0.01	
Gr 13	19.20	6.28	
	12.65	3.71	**5.33**
	21.9	6.00	
Gr 14	6.98	1.86	
	19.44	8.02	**6.30**
	22.50	9.01	

Figure 4. Mean of specific wear after sliding distance 0.72 km

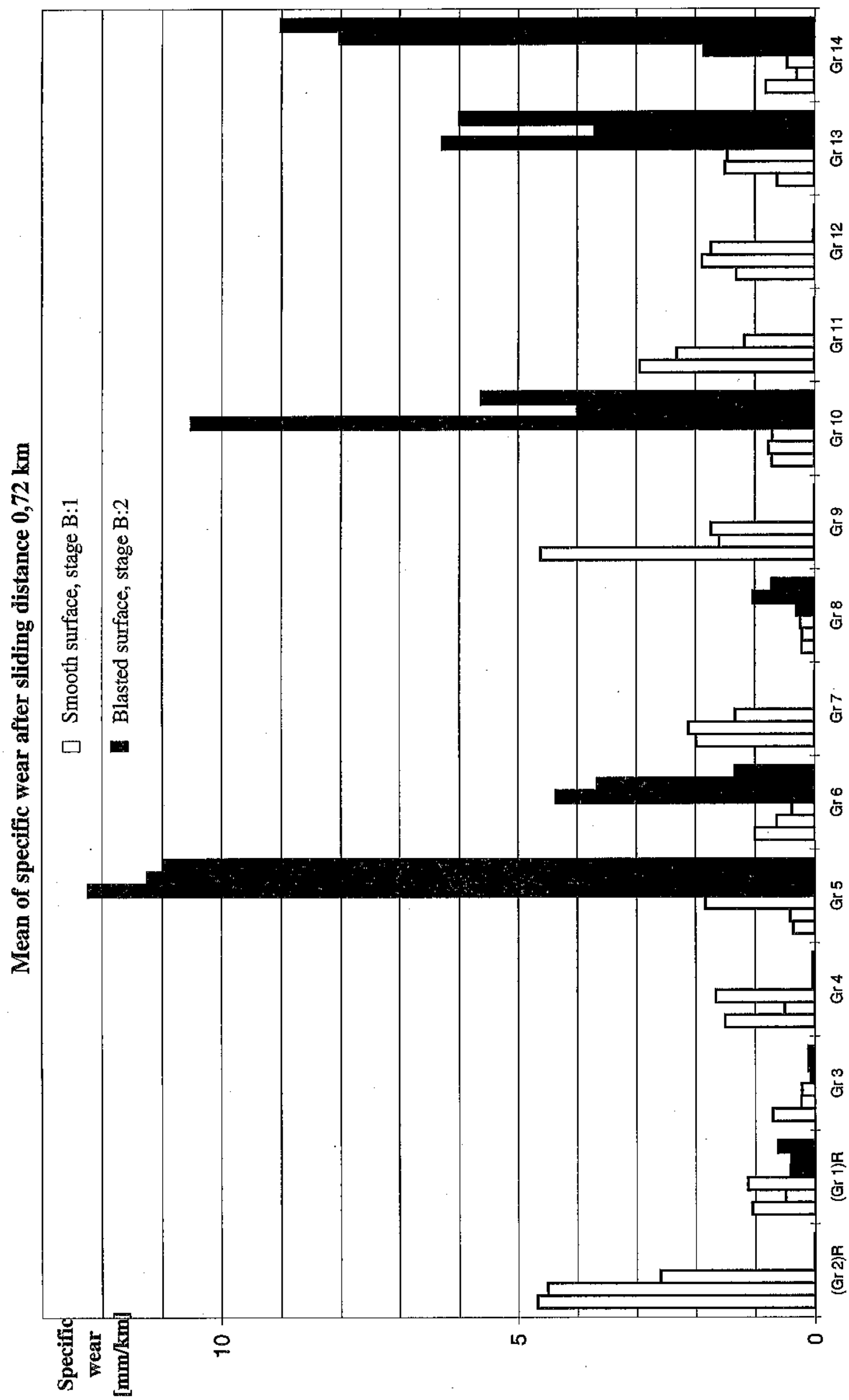
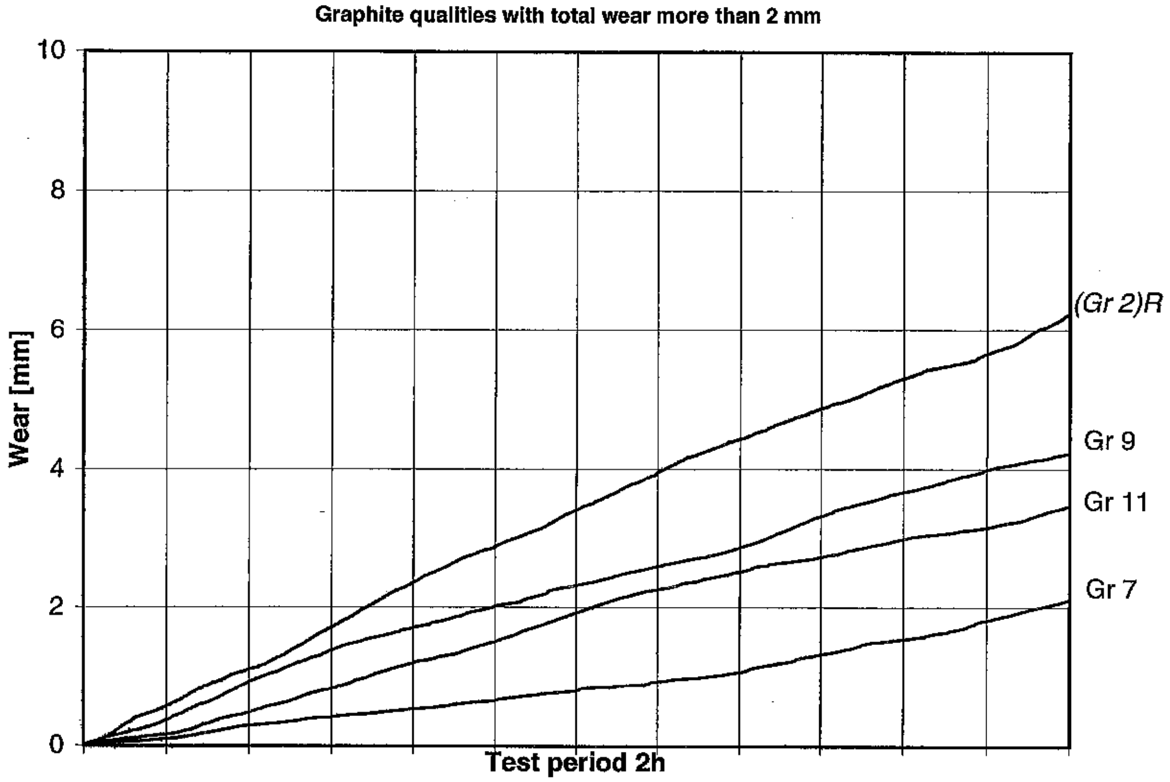


Figure 5. Wear history, smooth surface, addition 25 g AlO, stage B:1



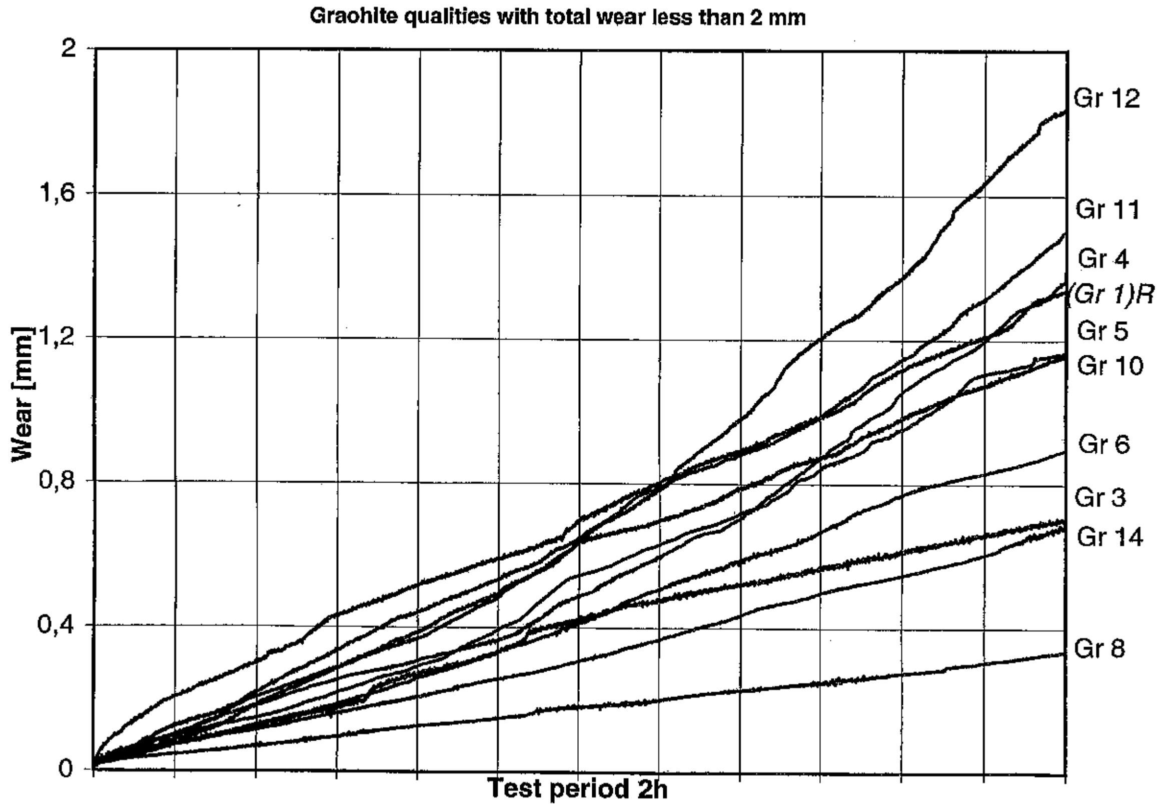
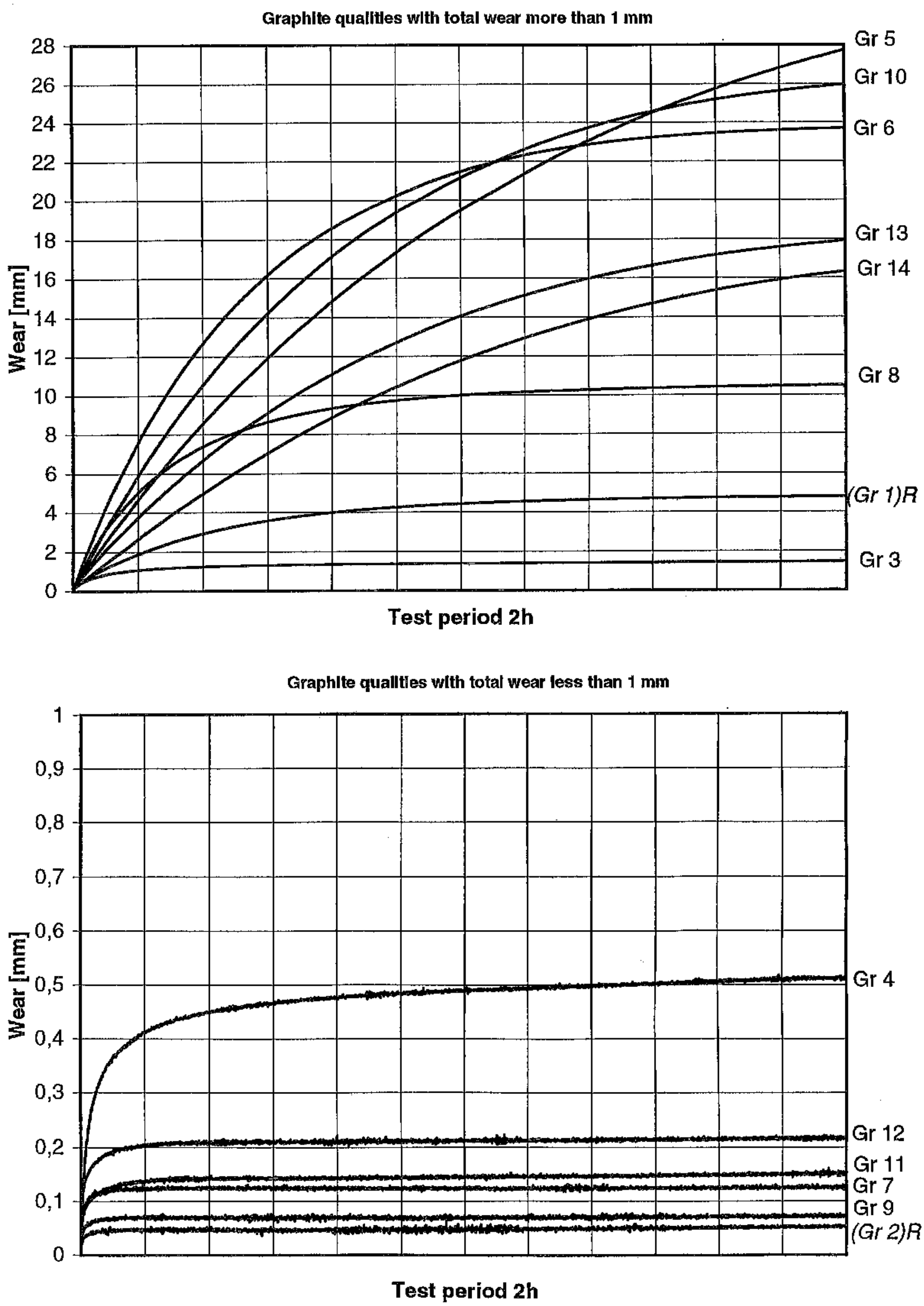


Figure 6. Wear history, blasted surface, stage B:2



7. Conclusions

The method described in the present report is based upon the use of established values of the mechanical parameters included in the tribo-system. These values have been determined in systematical pre-tests in stage A.

The results in stage B show that the developed method is applicable to distinguish and rank similar variants/qualities of a specific material with respect to wear (specific wear). It should be observed that one condition of the applicability of the method is that the . materials of the tribo-couple have very different hardnessess, where the test material is the softer.

Accordingly, as a result of the tests there could be established significant differences between the wear of the 14 qualities of graphite, which made the ranking of the qualities possible.

8. Acknowledgement

The financial support from Barsebäck Kraft AB, OKG and Vattenfall AB/Ringhals is gratefully acknowledged. I also wish to thank mr Börje Wegemar, ABB Atom AB, for valuable discussions and fruitful support of the experimental planning.

Special thanks go to my colleagues at SP, Lennart Aronsson and Mohammed Hoseini, for their testing work and evaluation and presentation of the results in this report.