

The Measurement of Sliding Friction and Wear at High Temperature

M. G. Gee & C. S. Matharu

National Physical Laboratory, Teddington, Middlesex, TW11 0LW, UK

1 INTRODUCTION

There is increasing interest in the use of ceramics in gas turbines and internal combustion engines. This is prompted by the attractive properties of ceramics such as their good high-temperature strength and low thermal conductivity which offer opportunities for reducing power losses and improving performance.

Because of the possibility of using ceramics for wear-resistant components in engines, there is a demand for information about the wear and friction of ceramics at elevated temperatures. However, before results from laboratory wear tests can be confidently used by engineers and designers, it is important to assess the relevance of these tests.

There have been a number of studies of the wear and friction of ceramics at moderate temperatures (up to about 800°C) (Aronov, 1987; Lankford *et al.*, 1987; Longson, 1983; Tomizawa & Fischer, 1986), where, with the addition of heating equipment, conventional wear test machines can often be used. However, there have only been a few reports of experiments at higher temperatures (Semenov & Katsura, 1979), and these are often not immediately relevant to engineering application because of the unrealistic geometry that has been used or because the tests have been carried out under high vacuum.

This paper describes equipment for performing wear tests in continuous sliding at temperatures up to 1500°C. The tests can be carried out in air, vacuum or other controlled atmospheres under applied loads of 5 to 100 N and speeds up to 1.5 m/s. The design of the ceramic specimens has been kept as simple as possible to reduce the costs of testing.

Some initial results of tests on reaction-bonded silicon nitride are described.

2 REQUIREMENTS

It is expected that ceramic components will be subjected to temperatures up to 1000°C in internal combustion engine applications, and up to 1400°C in gas turbines. However, as the applications are developed there are likely to be increases in the upper temperature limits of components. For this reason, an essential requirement for the test system was that it should have the capability for testing up to a temperature of 1500°C.

The geometry of the test system has a considerable effect on the magnitude and type of wear that is observed. Thus the results that are obtained using one test geometry and environment cannot immediately be applied to applications where the service conditions are radically different. The approach that was adopted in the current design was to choose a simple continuous sliding geometry which would allow a fundamental understanding to be developed of the processes that occur in high-temperature wear of ceramics. This knowledge should then be applicable to other geometries and types of motion.

Since oil-based lubricants cannot be used at high temperatures, the unlubricated wear of ceramics is of initial interest. However, this does not exclude the examination of solid self-lubricating coatings which have been developed recently (Corte & Sliney, 1987).

Some ceramics oxidise in air at high temperatures. This may have a considerable impact on wear and friction processes. In order that the effects of changes in atmospheric composition can be examined, it is important to be able to change the atmosphere surrounding the wear specimens. The easiest way of achieving this is to enclose the wear system in a vacuum enclosure, then evacuate and back-fill with the appropriate gas composition.

Ceramics are difficult and expensive to machine. In order that the cost of testing may be reduced, it is important to make the specimen geometry as simple as possible. In the test system described here, this was achieved by the use of a basic ring geometry which can easily be produced from most ceramics as an unmachined sintered body. Machining is then confined to simple grooving and grinding of the wear surfaces, thereby avoiding the machining of holes.

3 TEST SYSTEM

The general assembly of the test system is shown in Fig. 1. Most of the system was manufactured from stainless steel, with those components subjected to high temperatures made out of high purity alumina or silicon nitride. The control and signal conditioning electronics are housed in the rack mounted to the side of the system.

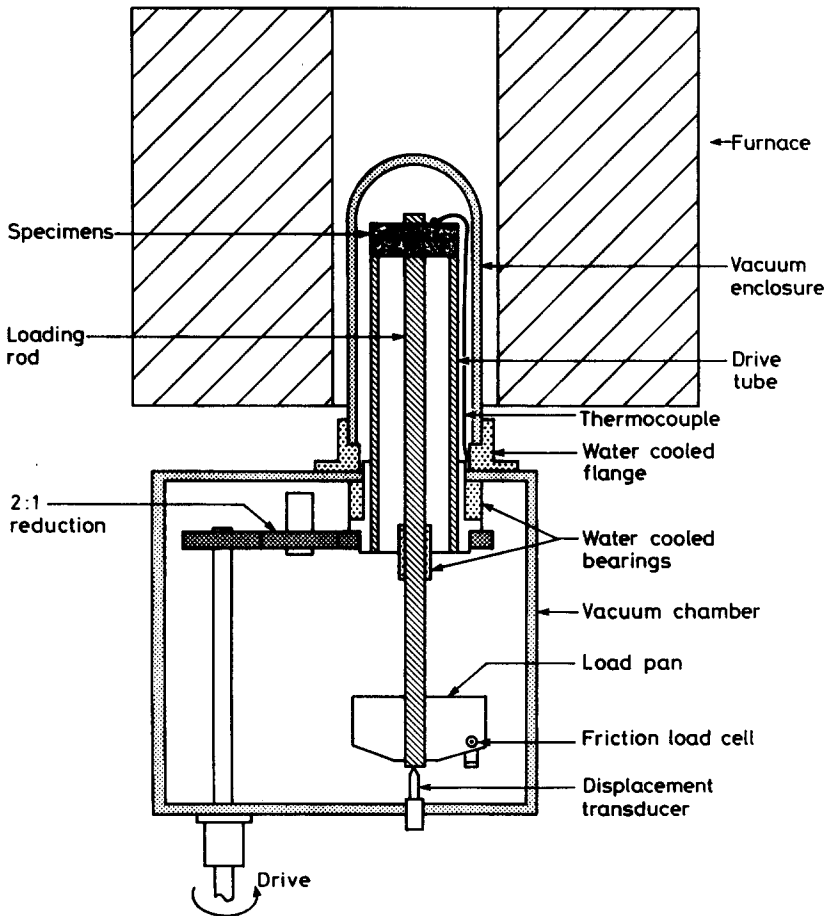


Fig. 1. General arrangement of test system.

3.1 Specimen geometry and drive

The specimens are rings with the flats of the rings used as the surfaces in contact (Fig. 2). The contact area between the surfaces can be reduced by machining most of the flat away from one of the rings to leave an annular surface in continuous contact, and can be machined away even more to leave three small areas in contact (this is broadly equivalent to a 'three pin on disc' geometry). The specimen assembly is shown in Fig. 3. The lower specimen is driven by a ceramic drive tube through a coupling piece which fits into straight slots cut in the lower face of the specimen. The upper specimen is kept stationary and is loaded by a central ceramic rod and driving pins.

The system is driven by a 0.37 kW DC variable speed motor. The drive is

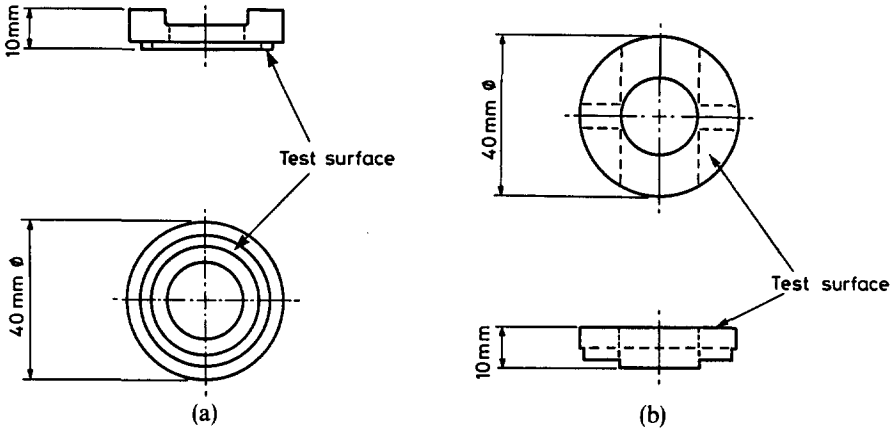


Fig. 2. Specimens: (a) upper, (b) lower.

transmitted to the ceramic tube by flexible couplings and a 2:1 reduction gear. To enable tests to be carried out in a vacuum or other controlled atmospheres, the drive is taken into the vacuum chamber through a coupling where the seal is achieved using magnetic fluid. The speed of the motor can be varied continuously from 0 to 800 rpm. The motor control incorporates a relay which cuts off power to the motor when the frictional torque exceeds a pre-set value (normally equivalent to a friction coefficient of 1.5).

3.2 Specimen heating and environmental control

The specimens are heated by a furnace which is mounted on an adjustable pedestal. This can be lowered on to the specimen assembly and locked into position. Two different furnaces are used: a wire-wound furnace with

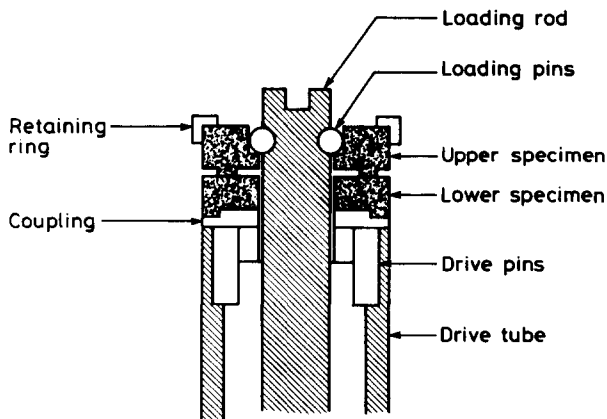


Fig. 3. Specimen assembly.

Kanthal windings for temperatures up to 1200°C, and a SiC element furnace for temperatures from 1000 to 1500°C.

The specimen temperature is measured by an armoured thermocouple led through the seal with the tip located in contact with the stationary specimen. The thermocouple output is monitored by a digital thermometer. The temperature of the furnace, which is monitored by a thermocouple buried in the windings of the furnace, is controlled by a proportional-integral-derivative (PID) device which controls the power supplied by a matched thyristor unit.

For tests in vacuum, a closed alumina tube is fitted over the specimen assembly and located in a water-cooled flange. Cooling water is provided via flowmeters mounted on one side of the support framework. The whole volume inside the enclosing tube and the gear chamber is evacuated, and can then be back-filled with gas of a known composition. To perform tests in laboratory air, the enclosing tube is not fitted. The vacuum system consists of a high-throughput diffusion pump backed by a rotary pump, giving a vacuum better than 10^{-2} Pa.

3.3 Measurement and loading

The load is applied by adding weights to a loading pan mounted at the base of the central loading rod. A load cell placed at one side of the load pan prevents its rotation and measures the frictional force. The load cell output was calibrated by known weights before it was installed into the system, and the accuracy of frictional measurements was checked by applying a known torque to the upper specimen with the load cell in position. No error was observed. The vertical displacement of the loading tray gives a measure of wear and is monitored by a displacement transducer. The sensitivity of the displacement signal to the specimen temperature was checked by heating a specimen assembly at a controlled rate and observing the displacement output. This was a linear increase of $0.027 \mu\text{m}/^\circ\text{C}$ over a temperature range of 0 to 900°C. The outputs from the load cell and displacement transducer are continuously recorded on a chart recorder during tests.

4 INITIAL RESULTS

Preliminary experiments were carried out on reaction-bonded silicon nitride (RBSN) which is a ceramic produced by nitriding a silicon powder compact, leaving a porous silicon nitride microstructure. Specimens were prepared with the dimensions given in Fig. 2 and the wear surfaces were lapped and polished to give a finish of $0.1 \mu\text{m}$ Ra (measured with a $250 \mu\text{m}$ cut-off

length). All tests were carried out with an applied load of 10 N and a speed of 0.1 m/s.

The normal test procedure was to assemble the specimens and driving train, lower the furnace over the specimen assembly and then heat the specimens to the required temperature before starting the motor. For tests in a vacuum, the closed alumina tube was placed over the specimens and the wear system was evacuated before the specimens were heated. This procedure was satisfactory apart from a test which was attempted on RBSN at 1400°C in air, where the specimens were found to have seized together when the motor was started.

Figure 4 shows the variation of displacement with sliding distance in laboratory air at temperatures from 21 to 1170°C. There was an increase in wear with temperature and sliding distance at temperatures up to 900°C. However, at 1170°C almost no change in wear displacement was recorded. Normally there was a relatively high rate of wear at the start of the test, followed by a reduced wear rate which continued for the remainder of the test. The displacement curve for the test at 900°C showed an additional region of increased wear rate at a sliding distance of about 700 m.

When wear rates were calculated from the mass loss of the specimens, results for specimens tested in air generally reflected the displacement results, with an increasing wear rate up to 900°C, but with a mass gain measured for the tests at 1140 and 1170°C, making the calculation of a wear rate impossible for these tests (Fig. 5). (It must be noted that these results are likely to have been affected by oxidation of the wear specimens, and must be treated with caution, since any oxidation products that remained on the specimen will have increased the specimen mass.) The equivalent values for RBSN tested in vacuum are also given and show higher wear rates than the

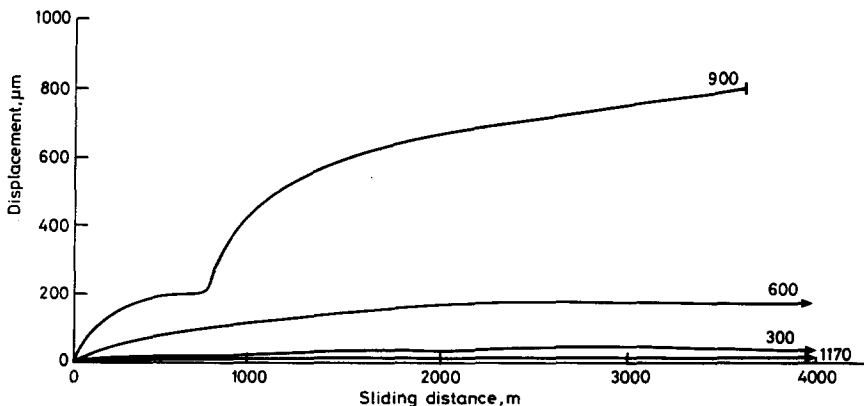


Fig. 4. Displacement traces for tests carried out in air. Numbers by traces are the temperatures of the tests.

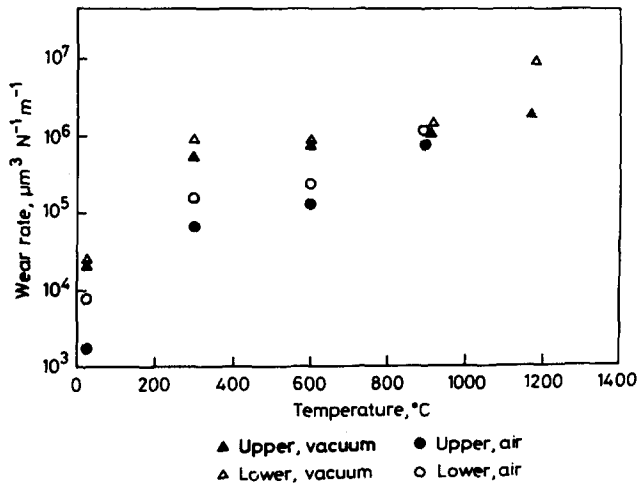


Fig. 5. Variation of wear rates calculated from mass losses with temperature.

tests carried out in air at temperatures up to 900°C, where the wear rates are nearly equal. It is interesting that the lower specimen always wore more than the upper specimen. This is particularly noticeable for the tests carried out in air.

The variation in friction coefficient (average value after initial period of test) with temperature is shown in Fig. 6. The friction coefficient was between 0.6 and 0.8 for specimens tested in vacuum at temperatures between 21 and 1140°C. This contrasts with the results for the specimens tested in air,

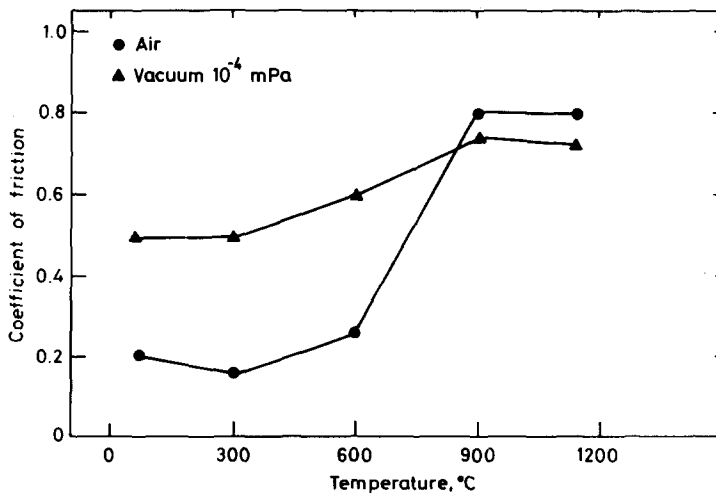
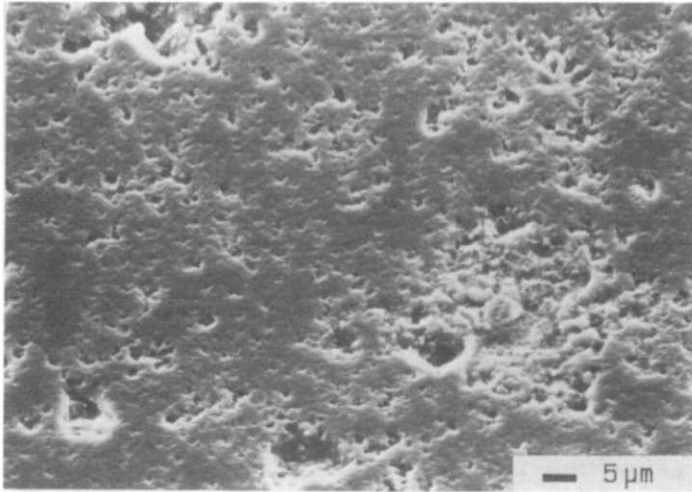
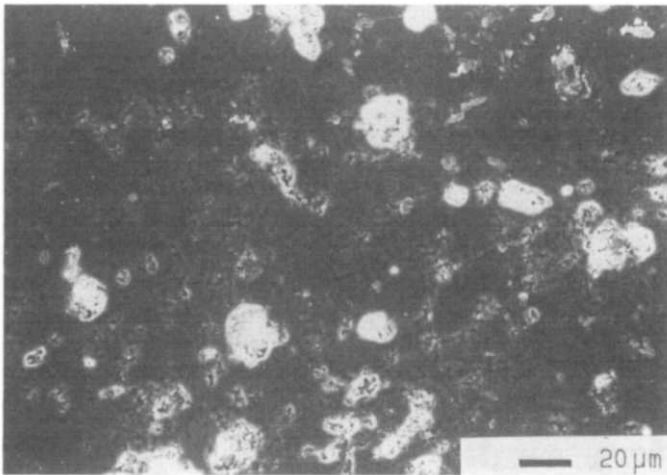


Fig. 6. Variation of friction coefficients with temperature.

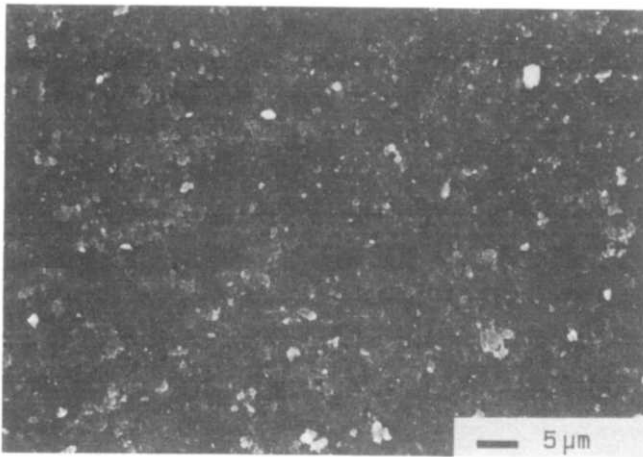


(a)

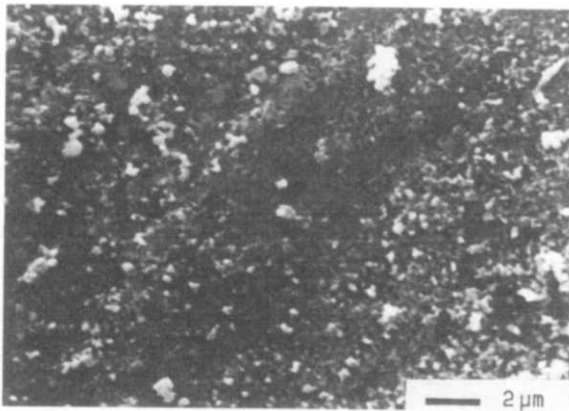


(b)

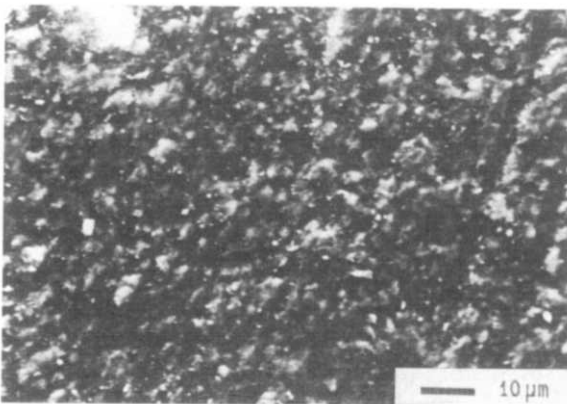
Fig. 7. Scanning electron micrographs of (a) untested specimen, (b) specimen heat-treated at 1140°C, (c) wear surface on upper specimen tested at 1140°C in air, (d) and (e) upper and lower wear surfaces of specimen tested in vacuum at 900°C.



(c)



(d)



(e)

Fig. 7.—*contd*

where the friction coefficient was quite low at about 0.3 up to a temperature of 600°C and then increased to about 0.7 at higher temperatures.

Examination of the wear surfaces in the SEM showed that the specimen tested in air at 1140°C is covered by layers of deformed material (Fig. 7). This was in contrast to the specimen tested in vacuum at 900°C where there was a considerable quantity of particulate material covering some parts of the surface, with other parts of the surface showing signs of grooving from abrasion processes. These wear surfaces were also compared with the unworn specimen surface which is featureless apart from some porosity. The surface of the wear specimen tested in air at 1170°C away from the wear area was covered by a layer of material formed by the reaction of the silicon nitride with the air.

5 DISCUSSION

5.1 Testing

In use, the test system proved to be reasonably easy and simple to operate. However, in some tests, a great deal of vibration was generated. Because of this, there was a tendency for the specimen assembly to fall apart during the test. This was cured simply by using high temperature alumina-based cements and platinum wire bindings to make the assembly more secure.

The measurement of wear displacement and frictional torque at the base of the system was also far from ideal, because of the long distance between the interface and the measuring transducers. Nevertheless, the calibration trials for the friction load cell and displacement transducer give confidence in the results, particularly with the low temperature coefficient of the displacement measurements of 0.027 $\mu\text{m}/^\circ\text{C}$; this is probably due to the fact that the central loading rod and driving tube are of similar lengths and made from identical materials, so that they form a balanced arm extensometer with approximately equal thermal expansions in the two arms (central rod and drive tube).

5.2 Effect of geometry and system

It is known from wear tests on alumina at room temperature (Gee & Almond, 1988) that the geometry of the specimens can have an important effect on the results. This can be due to many factors, such as whether the surfaces are in conformal contact or not, whether there is trapping of debris between the surfaces, and whether there are differences in the heat flow away from the wear interface.

In the preliminary tests carried out on RBSN, it is quite interesting that the lower specimen always wore more than the upper specimen. This is quite likely to be linked to the different geometry of the upper and lower specimens, with a raised annular ridge on the upper specimen pressed into a flat surface on the lower specimen. The effect of this ridge will be made clearer in tests which are in progress where specimens have been prepared with a flat surface on the upper specimen, and a ridge on the lower specimen.

It should be noted that the initial tests on RBSN were carried out with a planar contact between the two wear surfaces. Because there is quite a large clearance between the loading rod and the hole through the lower specimen, there is a possibility that the two specimens might not remain concentric. In practice this was not found to be true, with the final wear track on the lower specimen only a little wider than the ridge on the upper specimen.

To examine the effect of the continuity of the contact between the surfaces, tests will also be carried out on specimens where the continuous ring has been machined away to leave three equidistant raised areas in contact with the lower specimens.

It is expected that the dynamics of the test system will have a large effect on the results. An indication of the sensitivity of the system was the large amount of vibration that was generated in some tests and could be reduced or eliminated simply by touching the central loading rod quite lightly. This indicated that a major source is likely to be the torsional flexure of the central loading rod under the rapidly changing loads generated at the local contacts between the two surfaces. Further tests will be needed to investigate the detailed nature of the interaction of system dynamics with specimens.

5.3 Effect of other test conditions

An investigation of the effect of changes in other test conditions such as load, speed and the surface finish of specimens is also in progress. Initial results show that the wear rate is increased as the load is increased at a temperature of 900°C in air.

5.4 Initial results

Whilst a fully satisfactory interpretation of the results must await a more complete examination of the wear specimens and debris, it is clear that the oxidation of the reaction-bonded silicon nitride plays an important part in the wear that was observed. Thus the wear of RBSN in air is inhibited by the formation of layers of silica which separate the two surfaces, reducing the wear rates. At 900°C, the sudden increase in wear rate during the test was probably due to break-away of material from the wear surface, leaving a

fresh unoxidised surface which then oxidised, forming a new protective layer. The reason for the low apparent wear rates observed at 1140°C is not known but may be due to the softening of oxide forming, giving a higher degree of bonding between oxide and specimen and thus a greater retention of oxidised material which remains to carry the load.

The specimens tested in a vacuum exhibited a higher wear rate than those tested in air at temperatures up to 900°C where the wear rates were almost equal. A large volume of particles was generated, possibly as a product of the abrasion of one wear surface by asperities on the other surface, or in the later stages of wear by the wear debris trapped between the wear surfaces.

The low friction exhibited by the specimens tested in air at low temperatures is likely to be due to the reduced interaction of the wear surfaces because of the formation of intermediate layers of oxidised material.

6 CONCLUSIONS

A high-temperature wear testing system has been designed and built for wear tests on ceramics up to 1500°C. The specimen geometry is ring-on-ring and continuous sliding. Tests can be performed in air, under a vacuum, or in a controlled atmosphere.

The results of initial tests on a reaction-bonded silicon nitride show that the wear and friction results depend on the oxidation of the RBSN. There was an increasing wear rate with temperature for specimens tested in air up to 900°C, but only a small amount of wear at 1170°C in air. When a test was attempted at 1400°C in air the specimens seized together before the test could be started. Specimens tested in a vacuum gave higher wear rates than the specimens tested in air below a temperature of 900°C but the wear rates were nearly equal at this temperature.

High friction coefficients of about 0.6 to 0.8 were observed for specimens tested in vacuum at all temperatures, and above 600°C for specimens tested in air. Lower friction coefficients of 0.3 were observed for tests carried out in air below a temperature of 600°C.

REFERENCES

- Aronov, V. (1987). Wear resistance anomaly of magnesia partially stabilised-zirconia. *ASLE Trans.*, **30**(1) 100-4.
- Corte, C. D. & Sliney, H. E. (1987). Composition optimization of self-lubricating chromium-carbide based composite coatings for use to 760°C, *ASLE Trans.*, **30**(1) 77-83.

- Gee, M. G. & Almond, E. A. (1988). Wear testing for design, *Proceedings of the conference on materials and design*, May 1988, Institute of Metals, 23.1–23.6.
- Lankford, J., Wei, W. & Kossowsky, R. (1987). Friction and wear of ion beam modified ceramics, *J. Mat. Sci.*, **22**, 2069–78.
- Longson, B. (1983). Lubrication of high temperature ceramic materials, *Tribol Int.*, **16**(4) 221–5.
- Semenov, A. P. & Katsura, A. A. (1979). Investigation of friction and wear of corundum ceramics at temperatures to 1500°C, *Proceedings of the second international conference on the wear of materials*, April 1979, American Society of Mechanical Engineers, pp. 551–5.
- Tomizawa, H. & Fischer, T. E. (1986). Friction and wear of silicon nitride at 150°C to 800°C, *ASLE Trans.*, **29**(4) 481–7.