

REPORT

Some Methods for the Determination of the Strength of Oxides

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ABSTRACT

This report provides a brief summary of some possible techniques that could be employed to determine the high temperature strength and fracture strain of metallic oxides. The report focuses on three main methods, namely, (a) bend testing, (b) hot hardness, and (c) torsion testing. These techniques will be assessed as to their suitability for determining the properties of oxides that are required for process control and modelling relevant to the rolling and forging industries.

Project MMP6: Measurement of friction and heat transfer coefficients and strength of oxides for rolling and forging process control models.

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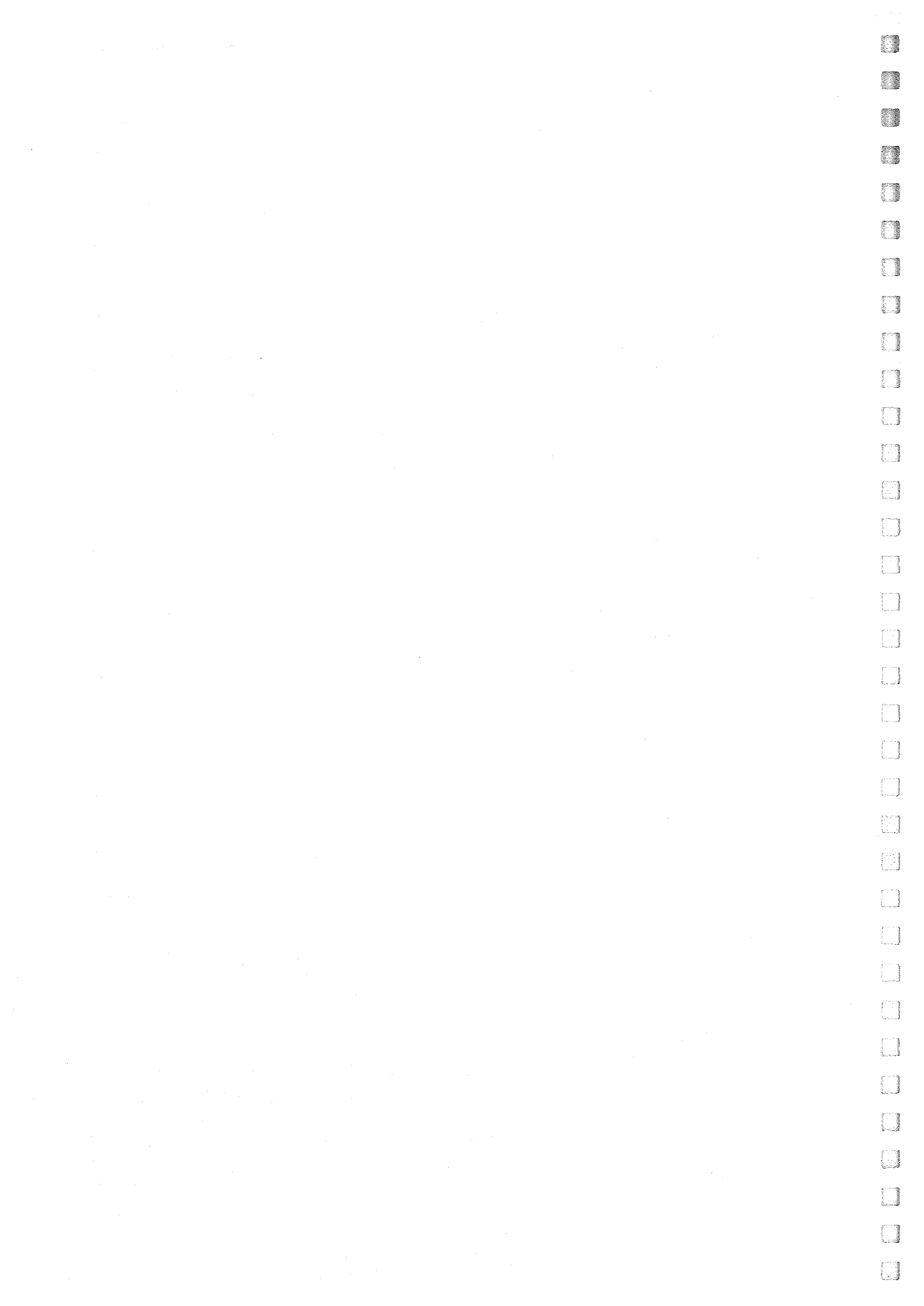
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Approved on behalf of Managing Director, NPL, by Dr C Lea,
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1 INTRODUCTION

It has been suggested that a knowledge of the strength and fracture strain characteristics of metal oxides is essential as input data for process control models for improving products produced by rolling and forging. As a consequence, a specific milestone was included in the Invitation to Tender for the DTI Funded Project MMP6: Measurement of Friction, Heat Transfer and Strength of Oxides. The NPL proposed to evaluate three methods to characterise the high temperature strength of oxides, namely

- (a) Bend testing
- (b) Hot microhardness
- and (c) Torsion testing

which are shown schematically in Figure 1.

These techniques were selected because of previous experience of the use of the techniques for the testing of metals, and it was anticipated that the methods could be adapted for the measurement of the high temperature strength and fracture strain of the oxides.

An oxide layer can significantly influence the interfacial heat transfer coefficient between a die and workpiece during forging, or the roll and feed stock during rolling. As deformation of the workpiece, or feed stock occurs, the oxide is also deformed, and if it breaks the underlying substrate becomes in direct contact with die or roll surface, resulting in a much higher heat transfer coefficient.

The integrity of protective oxide scales and coatings has been extensively investigated for materials subject to high temperature exposure during service for components in power plant or aerospace application, eg blades in steam or gas turbines. In these cases a knowledge of oxide strength or fracture strain is important for estimation of remnant life in components and indeed the possible methods for characterising the oxide layers have been reviewed elsewhere (see special issue of Materials at High Temperatures, Vol 12, Nos 2-3, 1994 "Mechanical Properties of Protective Oxide Scales - Selected papers of the workshop held at NPL, June 20-22 1994).

However, it should be appreciated that in general such methods have not been used over the higher temperature ranges that the materials are subjected to during metal working or processing. Hence the techniques may need some adaption to allow measurements to be carried out at higher temperatures, or on thicker oxide scales.

2 BEND TESTING

Four point bend testing is widely used in preference to three point bending because the stress distribution across the inner pair of rollers is uniform unlike that in the case of 3 point bending. The techniques for determining fracture strain data on oxides using bend testing has previously been reported by Nagl et al (1992), where techniques for measurements on equipment such as that schematically illustrated in Figure 2 are described. In essence, the oxidised testpiece is first prepared so that a raft of oxide is obtained which can be placed between the inner rollers such that it is strained in tension or compression - bottom or top side, respectively. A standard tensile test machine is used to apply the load to the bend jig. The strain to fracture is estimated by use of acoustic emission (AE) in which a waveguide attached to the sample transmits an acoustic signal originating in the sample due to oxide coating. (In previous work the specimen was sufficiently long that it acted as the waveguide).

If the tests are carried out at room temperature, the simplest preparation procedure is to expose the entire testpiece to the oxidising atmosphere and then carefully remove all unwanted oxide by grinding. Particular care should be taken to limit damage to the remaining oxide raft; in practice, it has been found that by using 600 grit SiC paper damage is confined to within 200-500 μm of the edge, and it has been demonstrated that these damaged areas do not act as crack initiators. At high temperature it is necessary to mask all of the testpiece except the area of interest. The masking agent should reduce the oxidation rate to about 10% of that of the unprotected material and should not contribute to (AE) signals. Several commercially available, high temperature stop-off paints have been evaluated, but the most successful are based on aluminosilicates, which show a degree of viscoelasticity at high temperature.

Tests carried out at room temperature used a standard jig made from metallic materials, but for the high temperature tests alumina was employed. Generally, the alumina parts were noisier than the metallic jig, and hence it was necessary to set higher threshold levels for the high temperature tests. The furnace arrangement used for this work is shown in Figure 4, in which it can be seen that long samples were used which protruded into the furnace door. The door was opened to allow the AE transducer to be clamped to the testpiece, and the transducer was cooled with an air stream and a steel plate was placed over the furnace entrance to eliminate radiant heat from the furnace. Usually, a small preload was applied so that testpiece and jig were in contact, and hence the crosshead movement could be used to calculate the applied strain (see later). However, the preload caused some deformation and allowance for this deformation had to be made. A further correction was necessary in cases where the coating deformed underneath the rollers during testing. A general view of the apparatus is shown in Figure 4a, and a close up view of the testing jig can be seen in Figure 4b.

The advantages of the four-point bend test using flat testpieces are:

1. tensile and compressive stresses can be applied without changing the dimensions of the testpiece or test jig;
2. it is easy to attach the AE transducer directly to the testpiece and thereby eliminate the need to add additional interfaces;
3. the use of flat testpieces eliminates uncertainty in the stress calculation that results from inward movement of the metal/scale interface or outward movement of the scale during oxidation when using round testpieces;
4. with flat samples the thermal and growth stresses create a two-dimensional stress field with the same stress components in the x and y directions, while the z component is zero unlike with round testpieces; and
5. models based on buckling can be used to describe compressive deformation, whereas oxides on round testpieces require a composite treatment of both the inner rod and outer tube of oxide which is not currently available.

The main disadvantages are:

1. the influence of edge effects present in both the x and y directions;
2. variations in bending stress with distance from the neutral axis which amount to $\pm 1\%$ and $\pm 3\%$ for 10 μm and 50 μm thick oxides, respectively.
3. a bending moment is introduced which promotes delamination for tests in tension and discourages buckling for compression tests; and

4. the necessity of defining a given area of material between the inner rollers can add some uncertainty to the tests and makes testing at high temperatures difficult because of the somewhat restrictive criterion of obtaining a masking agent which is effective in reducing oxidation and is acoustically quiet.

Error analysis

Strain values ϵ from the four-point bend test are given by

$$\epsilon = \frac{3ftd}{u(3s - 4u)} \quad (1)$$

where f is the crosshead speed in millimetres per second, t is the time in seconds, d is the testpiece thickness in millimetres, u is the distance in millimetres between the outer and inner rollers and s is the distance in millimetres between the outer rollers. The main error is in the measurement of time to failure as indicated by AE (see later). All other parameters can be measured accurately, such that they cause only an overall error of $< 1\%$. The error in the measurement of time is absolute, so that at higher strains this has a reducing effect on the overall error. Typical error analysis yields maximum and mean errors of 15% and 11%, respectively, for a strain of 0.14%, and 10% and 6%, respectively, for a strain of 0.28%.

Checks were made to establish the validity of equation (1) by the use of strain gauges, and up to about 0.3% (the elastic region for deformation of the substrate) there was agreement to within $\pm 2\%$ of the strain determined by use of the strain gauge. In the plastic region the calculated values diverged from the measured values, as indicated in Figure 5. Thus, in the elastic region equation (1) can be used, but in the plastic region deformation has to be checked using strain gauges (only possible for tests at room temperature) or by observing the curvature at the end of the test to see whether it is uniform, ie no localised yielding had occurred during the test (see later). Figure 6 is a typical set of results for fracture of iron oxide on mild steel at 550 °C and RT when testing in tension. Figure 7 shows typical fractures in plan and cross-section for tensile and compressive failures on mild steel and nickel, respectively.

It should be noted that in a comparison of 4 point bend test measurements with conventional tensile or compressive tests, Saunders et al (1994) concluded that "the conventional (tension/compression) tests were generally easier to carry out at higher temperatures since the use of coating could be avoided and the uncertainty over the applied strain is eliminated". However, at present it would appear that suitable fixings with the AE equipment for high temperature testing are not available in the UK. More recent work at NPL in which a dual AE detector together with appropriate software allows comparison of AE signals and thus eliminates "background" noise from ancillary equipment. This has greatly improved sensitivity of the tests at high temperatures. Also reducing specimen length and the use of waveguides has made the test system more flexible.

3 HOT INDENTATION TESTING

3.1 INTRODUCTION

Hot indentation is a method for the measurement of the high temperature deformation properties of materials which only needs very small sample sizes. The basic concept is that an indenter of known shape is pressed into the sample with a known load, and the size of the depression is measured after a set time, or is measured continuously if time dependent deformation information is required. There is also another variant of the technique called self-indentation which is suitable for very high temperature measurements where two

samples of known shape are pressed together under a known load, and the change in shape gives a measure of deformation.

The hardness of a material is a measure of its plastic deformation. The numerical value of hardness is related to the yield strength of the material, and for metals the approximate relationship $H \approx 3Y$, where H is the hardness and Y the yield strength, is a reasonable description (Johnson, 1985). However, for ceramics the relationship between hardness and yield strength is not so clear cut (McHolm, 1990). One of the expected outcomes for the study of oxide strength is to give further information on how the hot hardness of oxide scales relates to their tensile strength properties.

Although the concept behind hot indentation testing is extremely simple, there are some issues which require further particular attention and are discussed in the following text. A commented bibliography of literature of hot indentation testing is also given.

3.2 INSTRUMENTAL CONSIDERATIONS

3.2.1 Indenter furnace

There has been a considerable amount of experimental work carried out with microindentation systems which have been modified for hot indentation testing by the simple addition of a sample hotplate to the stage of the instrument without specifically heating the indenter. The results of tests carried out with this type of system always give rise to the suspicion that the temperature of the sample at the indentation position is reduced by the conduction of heat away from the sample into the indenter. This concern can be alleviated to some extent by the introduction of procedures to preheat the indenter by resiting it on the sample at a very small preload before the actual indentation experiment is carried out.

It is more secure to perform the tests on a system where both the indenter and sample are separately heated such that the sample and indenter are ensured to be at the same temperature (Everitt (1992)). However, this can be quite difficult to achieve because of the small size of the indenter.

3.2.2 Measurements

The main measurement that is made is normally the size of the indentation measured by optical microscopy. Thus for Vickers and Knoop indentations, it is the size of the indentation diagonals, d (mm), that is measured and is used to calculate the sample hardness through the formulae:

$$HV_p = 1.854P/d^2$$

and

$$HK_p = 14.229/d^2$$

where HV_p is the Vickers hardness measured with a pyramid indenter and HK_p is the Knoop hardness, and P is the applied load, (Kgf).

The measurements of indentation diagonals can either be made in situ in the test system if it is supplied with suitable microscope facilities, but often instrumental considerations dictate that optimum optical conditions cannot be achieved. In this case it is better to make a series of indentation experiments, and then to measure the size of the indentation in a high quality optical microscope after the sample has been removed from the test system.

Increasingly, measurements of the displacement of the indenter into the test sample are made continuously as the test is carried out. This requires the use of carefully selected displacement measurement probes to ensure that the requisite accuracy and precision can be achieved. Displacement measurements are most often made by measuring the movement of the indenter shaft. However, this limits the accuracy of the measurements due to the introduction of errors from thermal expansion mismatch. It would be preferable to measure the relative displacement between the sample and the indenter, but this is very difficult experimentally.

3.2.3 Computer control

Modern computer based data acquisition and control technology gives the ability to perform more sophisticated experiments than hitherto. The key to the success of this strategy is the control of indenter loading, which is normally conducted through the use of servo technology.

Computer control gives the ability of performing well defined loading cycles, or if displacement based feedback control is available, well defined displacement cycles can also be carried out.

3.2.4 Indenter material

There are two main types of hot indentation experiments. The first of these is self-indentation, Atkins & Tabor (1965), Atkins, Silverio & Tabor (1966), where two pieces of the same material are pressed together and the change of shape of the pieces is measured. This is particularly useful for more reactive materials at the highest temperatures where the more conventional hard indenters react with the test sample.

The second type of hot indentation experiment uses a hard indenter into a softer sample. Normally these indenters are made from single crystal diamond or sapphire. There is a particular problem with the use of diamond for some metals, eg Fe based alloys, which react strongly with diamond at temperatures above about 700 °C. When a hard indenter is used, it is often assumed that the indenter shape remains constant during the experiment, and the indentation retains the geometrical shape of the indenter after the experiment.

3.3 TYPES OF MEASUREMENT

3.3.1 Hot Hardness

Hot hardness is calculated from the size of indentations measured after experiments have been carried out. Although hardness is a measure of the instantaneous plastic deformation of a material, hot hardness is measured under conditions where significant time-dependent deformation can make a significant contribution to the size of the indentation during the time that the indenter is loaded against the sample in typical hot hardness experiments. This means that the control and timing of hot hardness experiments is particularly important.

3.3.2 Indentation Creep

In indentation creep, the size of an indentation is measured as a function of time under load, either by making a series of indentations with different dwell times, or by measuring the depth of the indentation as the time under load is increased. The increase in size of indentation is due to creep processes, but in normal indentation experiments with a constant indentation load, the contact pressure will decrease as the size of the indentation increases, making interpretation of results quite complex. For further information on methods for deriving creep deformation information from indentation experiments, the reader is referred

to Sargent & Ashby (1989), Li, Hershall, Hooper & Easterling (1991), and Roebuck & Almond (1982).

3.4 NPL TEST SYSTEM AND TEST CONDITIONS

3.4.1 NPL Test System

The NPL test system uses a twin furnace design with both sample and indenter assembly heated (Figure 8a). The test system is enclosed in a vacuum chamber. Load is applied to the indenter by means of a novel magnetic force transmission system. This has opposing FeNdB magnets on either side of the vacuum chamber wall. The force transmitted across the vacuum wall, which is unbroken, is increased or decreased by driving the outer magnet by a servo motor system (Figure 8b). The force applied in the indentation is measured by a load cell just above the indenter shaft, and the displacement of the indenter shaft into the sample is measured by a capacitance displacement probe.

A general view of the apparatus is shown in Figure 9.

All measurements are recorded, and control of the motor drive and hence loading achieved through a computer based data acquisition and control system.

3.4.2 Test Conditions

Measurements of the hot hardness of oxide scales on steel will be carried out as part of project MMP6. As the furnace windings for the hot hardness oxidise in air, it will be necessary to form the oxide scales by pre-treatment in air on samples which will then be transferred to the hot hardness system for the hot hardness measurements. The tendency for oxide scales to break away during cooling because of thermal expansion mismatch will be minimised by the use of slow cooling rates.

Three conditions of scale will be measured, with a nominal scale thickness of 50 μm in each case. The scale will be produced at temperatures of 600 °C, 800 °C and 1000 °C.

Hot hardness measurements will be carried out on a range of loads from 1 N to 10 N, and at temperatures from ambient to 900 °C (the upper test temperature limit for the NPL system). Measurements will be carried out with a 15 s dwell period.

4 TORSION TESTING

4.1 INTRODUCTION

Torsion testing of solid testpieces has been used for many years for determining flow stress data for process models, at high rates of strain for ductile materials - Ryan et al (1983), Ryan & McQueen (1990 a&b). One of the disadvantages of solid testpieces tested under torsion is that a radial stress gradient is established which can lead to difficulties in interpretation of the data for constitutive modelling. In contrast, thin wall tubular torsion testpieces provide a reasonably uniform biaxial stress state to be generated across the wall of the testpiece. Machines were developed at NPL in the 1970s, Figure 10, and manufactured commercially, for high temperature torsion testing, which were extensively used for evaluating the growth of grain boundary cavities under multiaxial stress during creep (Dyson et al, 1976). It is proposed to modify one of these testing machines to evaluate shear stresses in oxide layers. It is proposed to utilise a testpiece that is machined in two pieces which are brought together using a loading train surrounded by a muffle furnace. An oxide layer, similar to that encountered in industrial rolling or forging operations, will be grown across

the interface of the two part testpiece under zero load, and subsequently subjected to a shear force in a controlled and monitored manner.

4.2 TORSION TESTING MACHINE

4.2.1 Testpiece Design

Two possible testpiece configurations have been considered which could be used to determine the shear stress of the oxide:

(a) An annular flat plate (Figure 11a)

This geometry would comprise an upper and lower flat annular flat plate which could be brought into contact and an oxide layer grown at the interface which would be sheared by the application of a load. Although relatively easy to machine, the testpiece has the disadvantage of having a radial stress gradient since it would not be practical to reduce the radial width of the annulus sufficiently to minimise the stress gradient, whilst providing an adequate surface area in contact to allow the load to be large enough to reduce the load measurement uncertainty to an acceptable level. In addition, it was considered that it would be difficult to ensure the setting up of the testpiece in the loading train to adequate precision and establishing the two faces to be sufficiently parallel.

(b) The spigot/tube testpiece (Figure 11b)

This geometry is considered the preferred option since in principle it should be easier to align the loading train, and the annular stress distribution will be significantly less. By undercutting the outer tube within the grip it will be possible to clearly define the axial gauge length of the oxide scale of the interface, although an allowance will need to be made for the thermal expansion of the testpiece at the various test temperatures.

Setting up of the testpiece and loading train will be of crucial importance but may it be argued that although any small lack of concentricity of the spigot and tube will result in a non-uniform annular oxide layer, since the applied forces will be distributed around the complete annulus, the resulting shear force will be an average across the mean thickness of the oxide layer. Testpieces will be machined in accordance with the drawing shown in Figure 12 having different annual clearances to allow different thickness of oxides to be evaluated.

4.2.2 Loading train

The torsion machines were originally designed for applying a twisting force to rigid tubular testpieces. Static forces could be applied either in a clockwise, or an anti-clockwise, direction under monotonic or cyclic loading. The predetermined load was applied to scale pans connected via flexible wires running over small pulleys and connected to a large horizontal wheel connected to the loading train, thereby allowing a torque to be applied to the testpiece. For creep testing, loads that caused a force less than the yield stress of the testpiece were used, however, the machine loading train and frame are sufficiently robust to allow forces greater than tensile strength of the material to be applied, so that plastic yielding of the thin wall testpiece at room temperature can be achieved.

Since the loading bars and testpiece comprised an integrally rigid leading train, it allowed specially designed flexible universal joints to be accommodated at either end of the train which allowed expansion of the train and provided self alignment. Since it is now proposed to substitute a two part testpiece, which is, of course, inherently non-rigid, for the solid tubular testpiece, it will be necessary to modify the design of the loading train. It will be necessary to provide external guides to the lower loading bar to prevent it falling over

sideways, whilst retaining the lower flexible alignment joint to allow for expansion. The upper loading bar will be replaced by a solid rod rigidly clamped to the top of the machine. Provision will be made to allow this upper rod to be initially adjusted to ensure axial alignment to the lower loading bar.

To monitor the torsional load, it is proposed to incorporate a torque load cell into the lower loading bar below the furnace.

A schematic diagram of the testing apparatus is given in Figure 13.

Weights will be put on the scale pan to provide a force in excess of the oxide shear strength. The weights/scale pan are to be supported on a motorised jack or pneumatic supporting strut, which can be lowered in a controlled manner thereby applying a twisting force to the testpiece. The rate of loading will be dictated by the speed of the jacking system.

4.2.3 Heating arrangement

The torsion machines at present incorporate a long three zone muffle furnace, which can be raised to provide access to the testpiece. Because of the new requirement of providing supporting guide roller to the lower loading train, it will be impractical to use the existing furnace, so it is proposed to replace it with a split furnace which can be mounted from one of the four supporting columns of the machine, and may be pivoted to swing horizontally to enclose the testpiece. Using the split furnace will considerably ease the difficulty of setting up the testpiece and attaching thermocouples, etc. The new furnace is a Kanthal wound three zone furnace, which incorporates sight holes and was previously used on a servo electric compression machine. It will be capable of heating up to 1050 °C, and the zones are independently controlled to allow an acceptable uniform temperature to be achieved along the testpiece gauge length.

4.2.4 Monitoring systems

4.2.4.1 Load

The applied torque will be recorded and monitored using a strain gauged load cell incorporated into the lower loading bar. Since the total annual twist of the testpiece will not be large, eg less than 90°, it will not be necessary to incorporate slip rings, and the electrical signals will merely be supplied and recorded via thin trailing wires. A simple calibration of the torque load cell can be carried out in situ in the testing machine by using a solid testpiece and by applying known weights to the freely hanging scale pan.

4.2.4.2 The annular rotation of the testpiece will be recorded via a rotary transducer connected to the bottom of the lower loading bar. At present a rotary LVDT is mounted on the machine, but a higher precision rotary capacitance transducer is also available.

It may be necessary to undertake a high temperature in situ calibration using a solid testpiece to determine the magnitude of the elastic compliance of the loading train which may need to be taken into account when calculating the rotational strain experienced by the oxide layer.

4.2.4.3 Temperature

The temperature will be determined by Pt-Pt/13%Rh thermocouples attached to the upper and lower parts of the testpiece, and monitored using the creep laboratory data logger. The thermocouples will be calibrated by comparison with an NPL Certified Reference thermocouple, following the procedure given in Creep Laboratory Manual (NPL Quality Procedure QPCMMT/B/107, Calibration of temperature monitoring system in the creep laboratory).

4.2.5 Testing procedure

A full detailed testing procedure will need to be written in due course to comply with the NPL's ISO 9000 Quality Procedure. However, in essence, the testing will comprise:

- (a) pre-alignment of the loading bars
- (b) installation of the tube/spigot testpiece
- (c) fine adjustment of the lower loading bar to ensure concentricity
- (d) attachment of thermocouples
- (e) positioning of the furnace and packing with thermal insulation
- (f) heating of the testpiece for a predetermined time and temperature to ensure the oxide has fully grown across the testpiece interface
- (g) application of the load at the required rate
- (h) recording of the stress/strain curve
- (i) analysis to determine the shear stress and fracture strain of the oxide.

5 TESTING PROGRAMME

It is proposed to evaluate the three techniques outlined above by undertaking a limited testing programme using a steel

It is intended to use similar testing temperatures and loading rates, if practical, on oxides of similar thickness, and a comparison will be made of the results from the three test methods.

At this stage it is not proposed to investigate aluminium or nickel based alloys, since in general, the influence of oxides in the production processes is thought to be less important compared to steels.

6 CONCLUSION

Using the test methods outlined in this report it should be possible to determine the strength and fracture strains of oxide films with an adequate precision, suitable for the generation of data for inclusion in process modelling and control software for rolling and forging.

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8 REFERENCES

8.1 REFERENCES CITED IN TEXT

Atkins, A G and Tabor, D (1965). "Materials Indentation Hardness for Use at Very High Temperatures". British Journal of Applied Physics 16, 1015-1021.

Atkins, A G; Silverio, A and Tabor, D (1966). "Indentation Hardness and the Creep of Solids". J. Inst. Metals 94, 369-378.

Dyson, B F; Loveday, M S and Rogers, M J (1976). "Grain Boundary Cavitation Under Various States of Applied Stress". Proc. Roy. Soc. A349, 245-259.

Everitt, N M (1992). "Indentation Creep on Single Crystal Magnesium Oxide". J. Hard Materials 3, 269-284.

Johnson, K L (1985). Contact Mechanics, Cambridge University Press.

Li, W B; Henshall, J L; Hooper, R M and Easterling, K E. "The Mechanisms of Indentation Creep". Acta Metall Mater. 39 (1991) 3099-3110.

McColm, I J (1990). Ceramic Hardness, Plenum.

Suzuki, T and Ohmura, T (1996). "Ultra-micro indentation of silicon at elevated temperatures". Phil. Mag. A, 74, 1074-1084.

Nagl, M M; Evans, W T; Saunders, S R J and Hall, D J (1992). Materials Science and Technology, 8, 1043-

Rees, D W A; Brown, M W; Hyck, T; Lohr, R D; Morrison, C J and Shammass, M (1992). "A Code of Practice for Torsional Creep Testing of Tubular Testpieces at Elevated Temperatures". Chapter 16, in Harmonisation of Testing Practice for High Temperature Materials. Ed. M S Loveday and T B Gibbons. Publ. Elsevier Applied Science.

Roebuck, B and Almond, A E. "Equivalence of Indentation and Compressive Creep Tests on a WC/Co Hardmetal", J. Mat. Sci. Letters, 1 (1982) 519.

Ryan, N D; McQueen, H J and Jonas, J J (1985). Can. Metall Quarterly, 22, 369-378.

Ryan, N D and McQueen, H J (1990). "Work Hardening Strength and Ductility in the Hotworking of 304 Austenitic Stainless Steel". High Temp. Technology, 8 (1), 27-44.

Ryan, N D and McQueen, H J (1990b). "Comparison of Dynamic Softening in 301, 304, 316 and 319 stainless steels". High Temperature Technology 8 (3), 185-199.

Saunders, S R J; Nagl, M M and Schutz, M (1994). Materials at High Temperatures, 12, 2-3, 103-109.

8.2 FURTHER BACKGROUND READING ON HOT HARDNESS

Atkins, A G and Tabor, D. Mutual Indentation Hardness Apparatus for Use at Very High Temperatures, Brit J Appl Phys, 16 (1965) 1015-1021.

[Classic paper giving foundations of hot indentation.]

Atkins, A G; Silverio, A and Tabor, D. Indentation Hardness and the Creep of Solids, J Inst Metals, 94 (1966) 369-378.

[Classic Paper.]

Brookes, C A; Brookes, E J; Howes, V R; Roberts, S G; Waddington, C P. "A Comparison of the Plastic Deformation and Creep of Type I, Type II and Synthetic Diamonds at 1100 °C under Conditions of Point Loading", J. Hard Mater. 1 (1990) 3.

[Soft indenter on diamond. Indenter heated.]

Everitt, N M. Indentation creep on single crystal magnesium oxide, J Hard Materials, 3 (1992) 269-284.

[Dual furnace system. Found that indenter heating was essential.]

Green, P and Brookes, C A. "Hardness, Friction and Wear of Engineering Ceramics in the Temperature Range 20-1500 °C". J. Hard Materials, 3 (1992) 169-183.

[Hot hardness measurements on various ceramics. Both indenter and sample heated.]

Gupta, Das S; Jacobs J K, King H W and Payzant, E A. Measurement of Hot Hardness by Diamond Pyramid and Mutual Indentation, Proc 13th Plansee Seminar, Eds H Bildstein and R Eck, Metallwerk Plansee, Reutte (1993) Vol 4, pp 381-387.

[Emphasises depth sensing; computer control; mutual indentation; measurement on alotic (alumina-TiCN).]

Hirai, T and Niihara, K. Hot Hardness of SiC Single Crystal, J. Mat. Sci. Letters, 14 (1979) 2253-2255.

[Measure hot hardness of SiC using Nikon system.]

Morrell, R; Byrne, W P; McCormick, N J; Reece, M J; Gee, M G; Blackburn, S; Greenwood, P and Senhenn, P G. "The hot hardness of ceria partially stabilised zirconia", Euro-Ceramics, Volume 3, Eds. G de With, R A Terpstra and R Metselaar, Elsevier Science, (1989) pp 3.346-3.350.

[Hot hardness measurements on ceria stabilised zirconia using dual furnace system.]

Sargent, P M and Ashby, M F. "Indentation Creep", Cambridge University Engineering Department Report CUED/C-mats/TR.145 February 1989.

[Survey of literature on indentation creep and methods of data analysis.]

Silva, R F and Vieira, J M. Hot Hardness of Si₃N₄ Based Materials. J. Mat. Sci., 30 (1995) 5531-5536.

[Stress state under indenter is a hydrostatic highly confirmed compression combined with a localised, large shear stress. Oxford dual furnace system.]

Suzuki, T and Ohmura, T. Ultra-microindentation of silicon at elevated temperatures, Phil Mag A, 1996, vol 74, 1073-1084.

[Nanoindentation of silicon at temperatures up to 600 °C. Also note that temperature drop due to lack of indenter heating was only 1-2 °C by thermocouple experiments. Attributed this to heating of indenter by radiation when held above sample.]

9 FIGURES

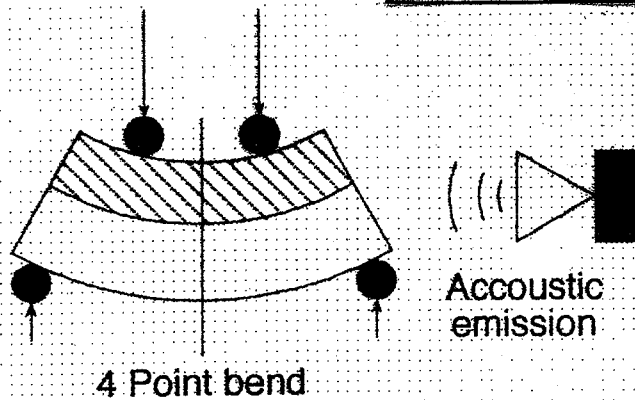
- Figure 1 Schematic diagram of three test methods for the characterisation of oxides.
- Figure 2 Schematic diagram of four point bend test rig.
- Figure 3 Diagram of the equipment used for four point bend testing at elevated temperatures.
- Figure 4a General view of four point bend test rig on testing machine.
- Figure 4b Close up of four point bend test rig.
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- (a) an annular flat plate
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MMP6

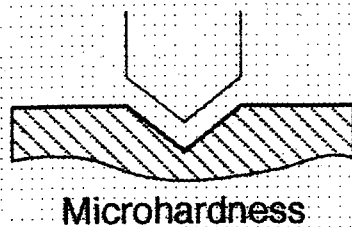
MEASUREMENT OF INTERFACIAL PROPERTIES
FOR
ROLLING, FORGING & EXTRUSION

Milestone M6

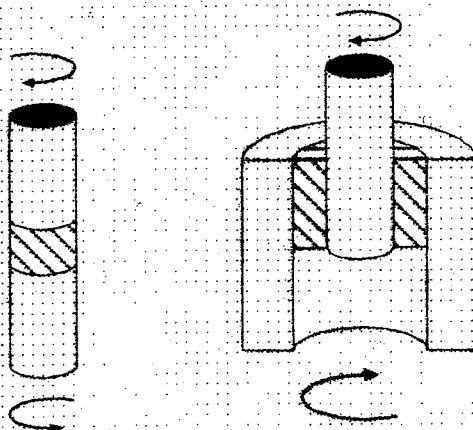
STRENGTH MEASUREMENT OF OXIDES
AT HIGH TEMPERATURES



Dr. Stuart Saunders



Dr. Mark Gee



Mr. Malcolm Loveday

Figure 1 Schematic diagram of three test methods for the characterisation of oxides.

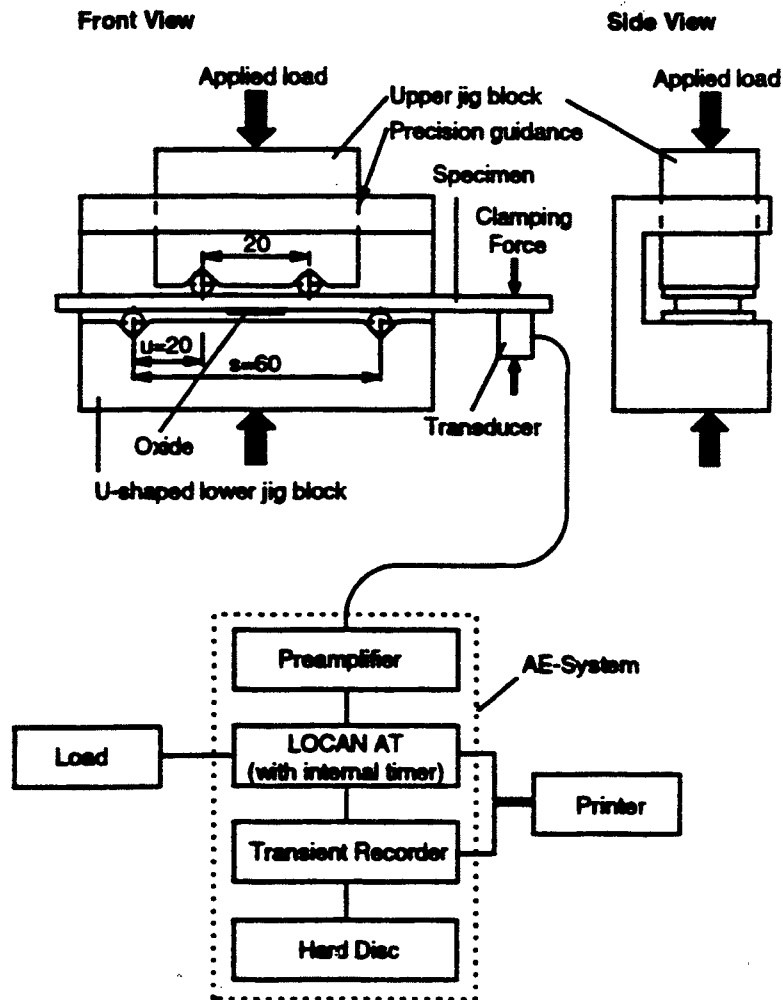


Figure 2 Schematic diagram of four point bend test rig.

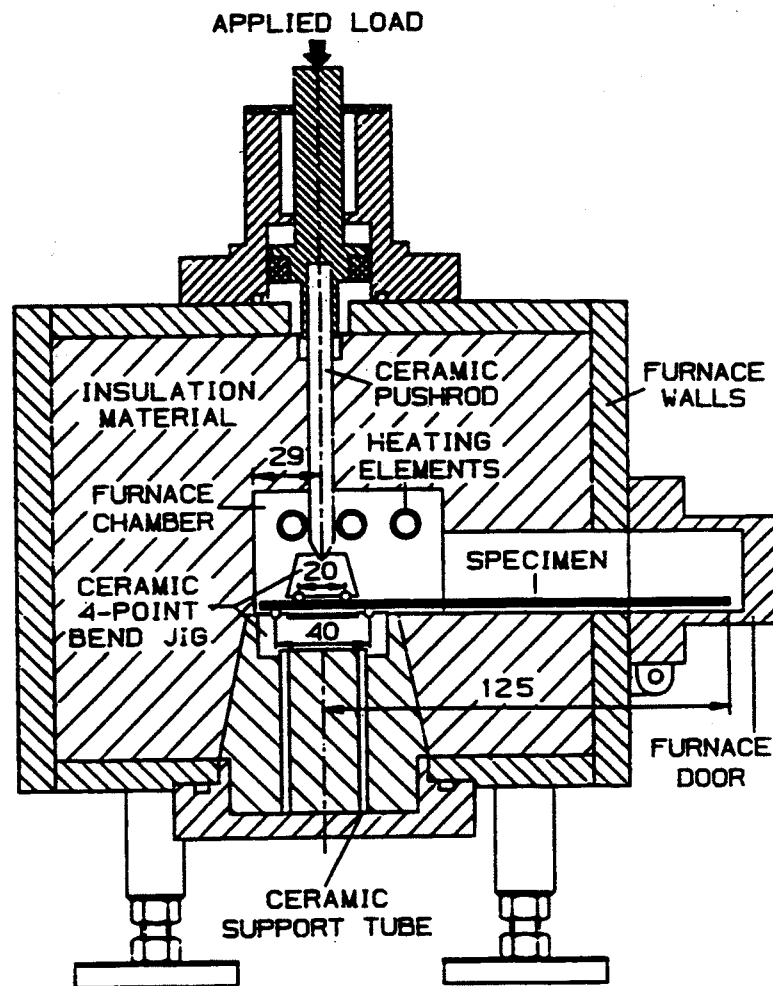


Figure 3 Diagram of the equipment used for four point bend testing at elevated temperatures.

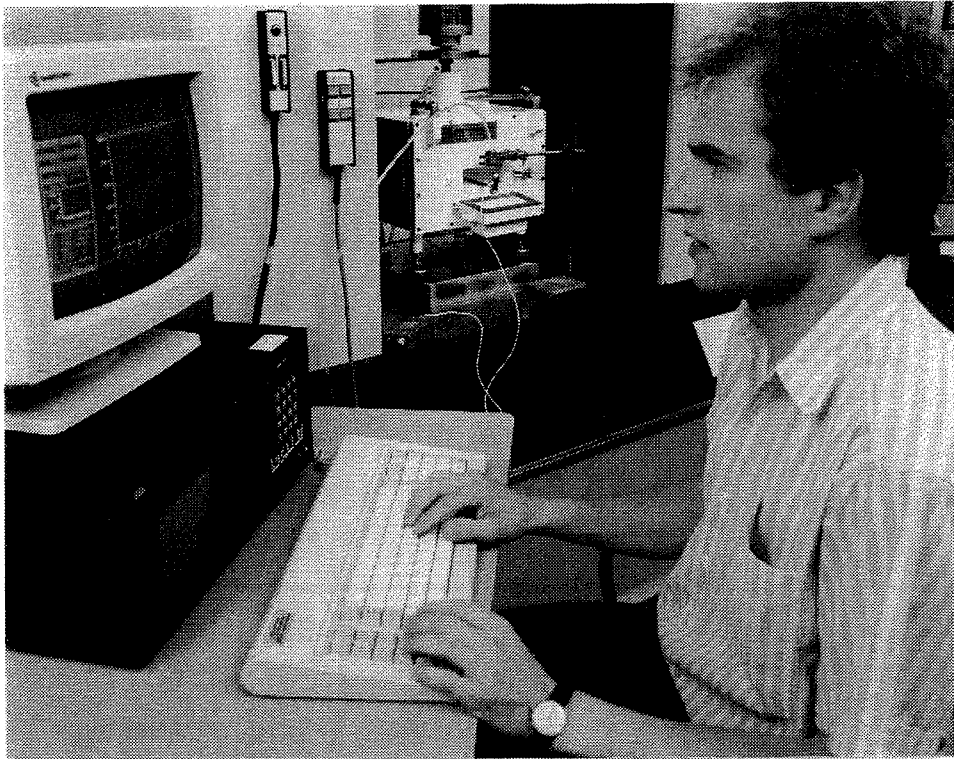


Figure 4a General view of four point bend test rig on testing machine.

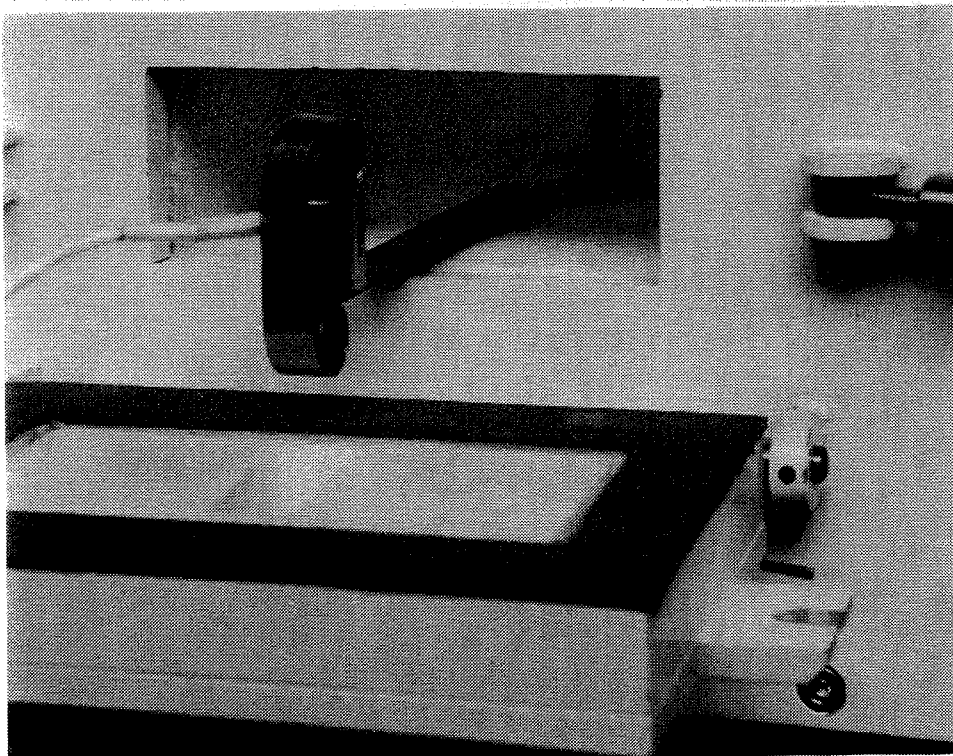


Figure 4b Close up of four point bend test rig.

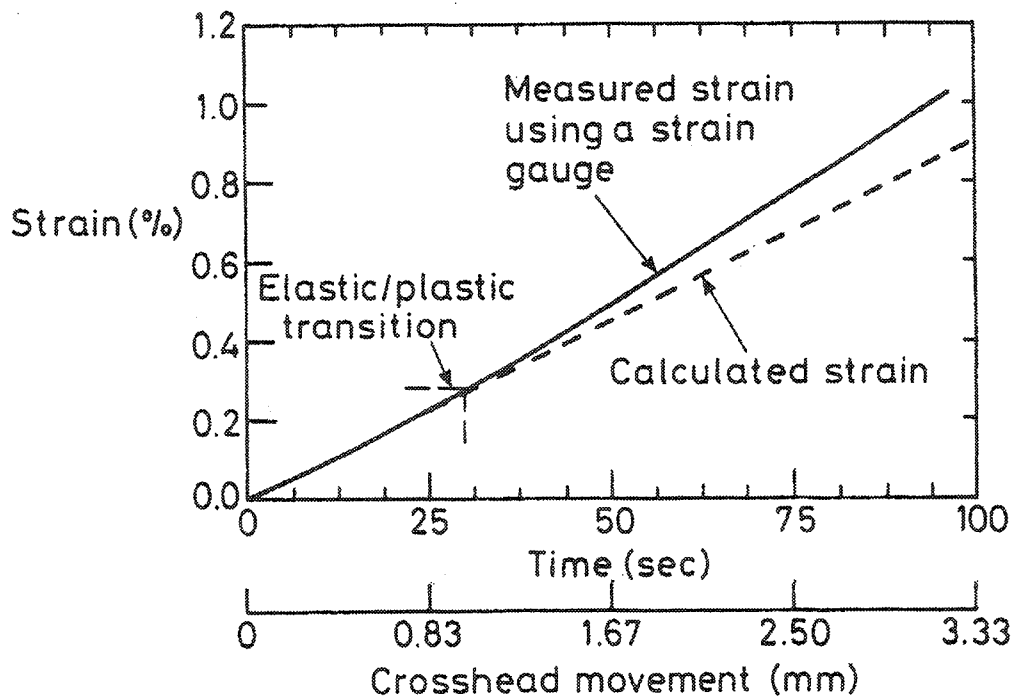


Figure 5 Errors in strain measurement from crosshead movement.

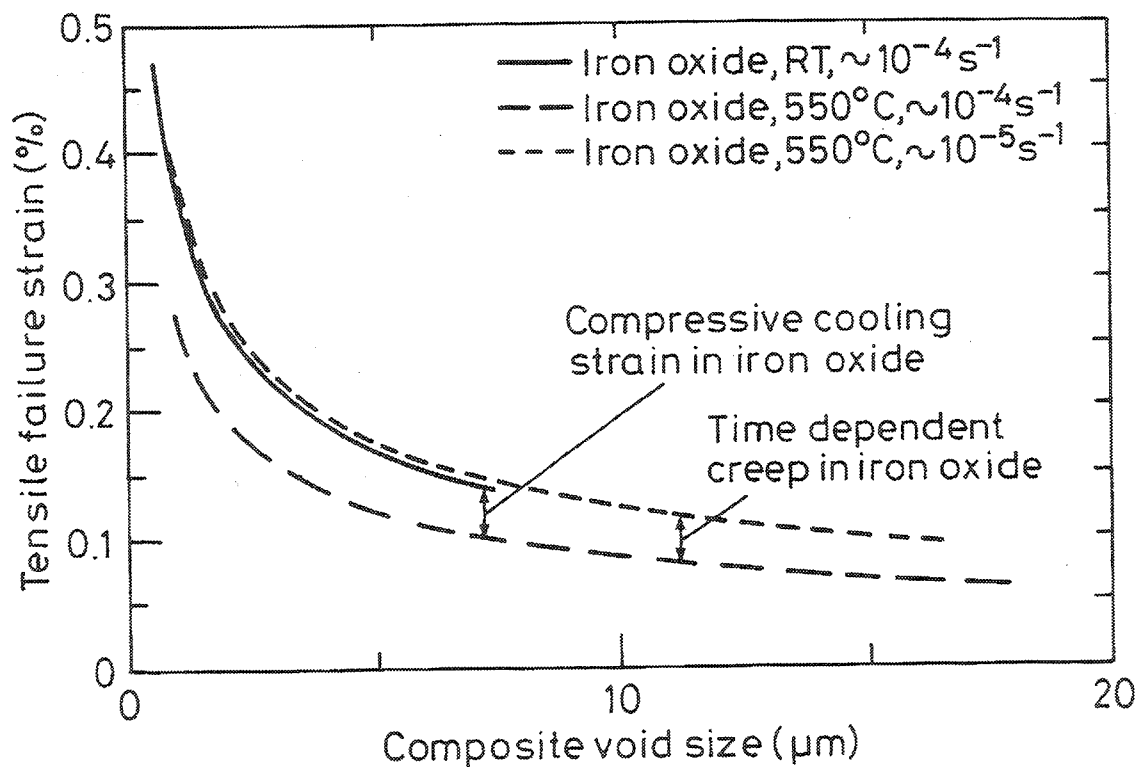
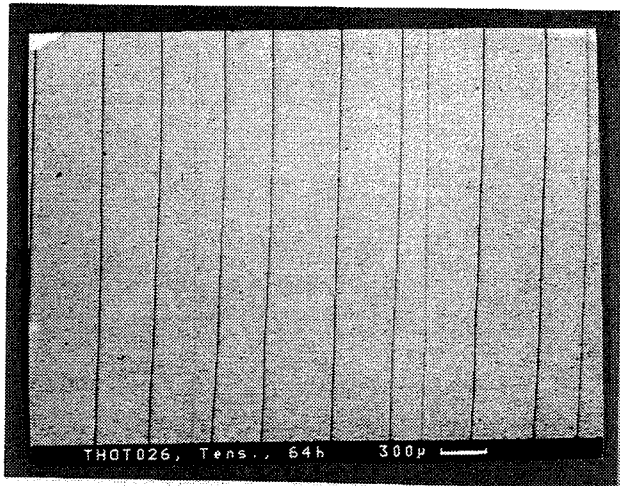
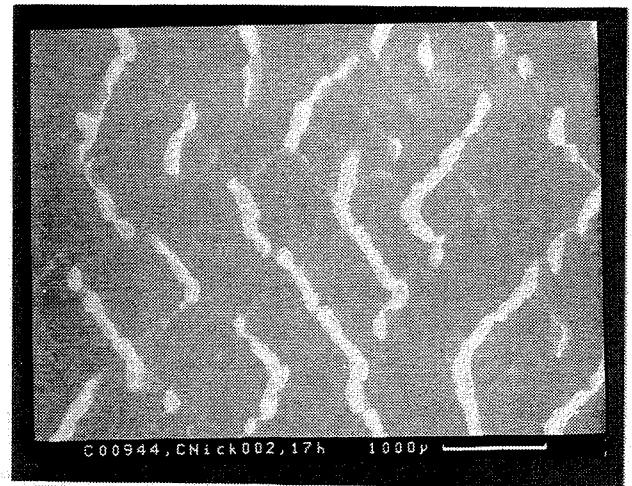


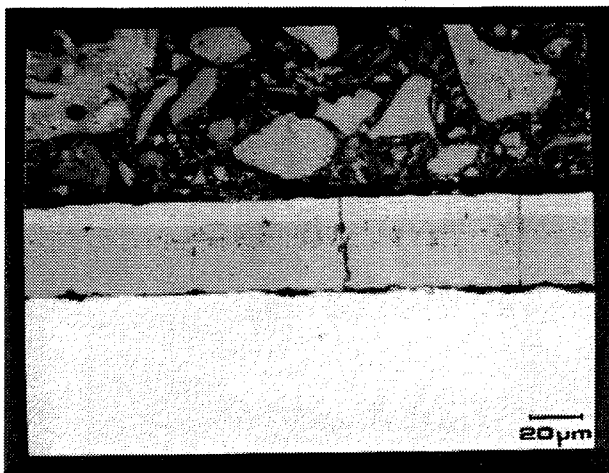
Figure 6 Tensile fracture strain of iron oxide as a function of defect/void size for two strain rates and temperatures.



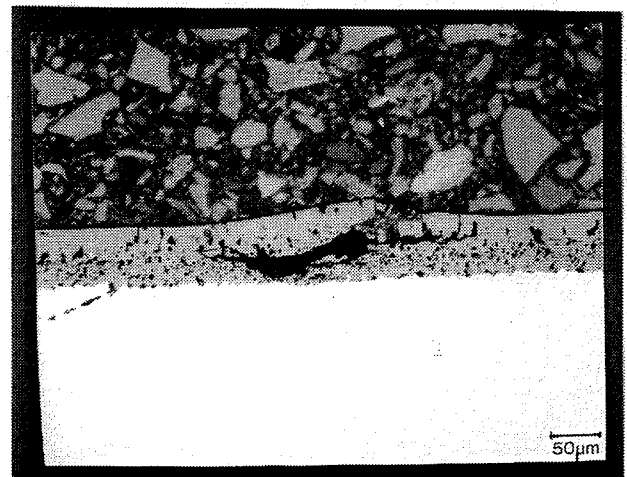
7a Plan view of tensile failure (mild steel)



7b Plan view of compressive failure (nickel)

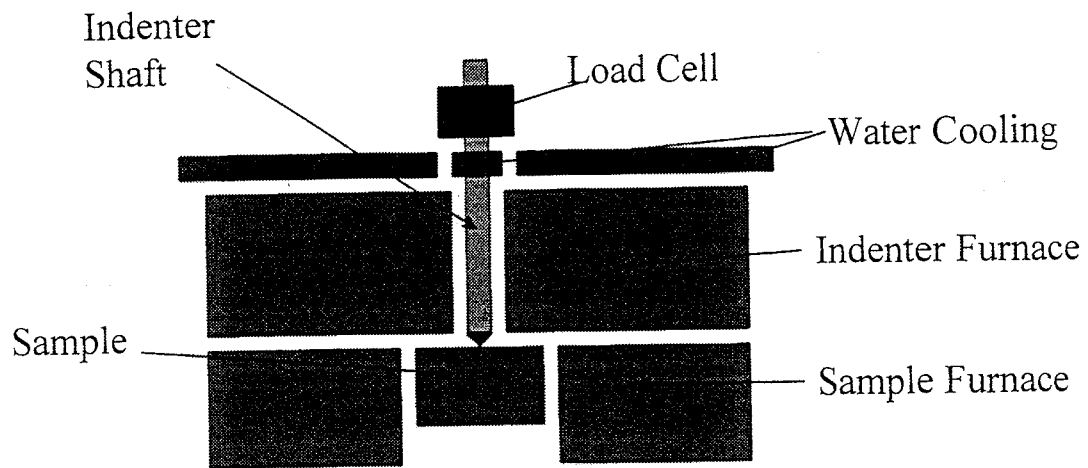


7c Cross section of tensile failure (mild steel)

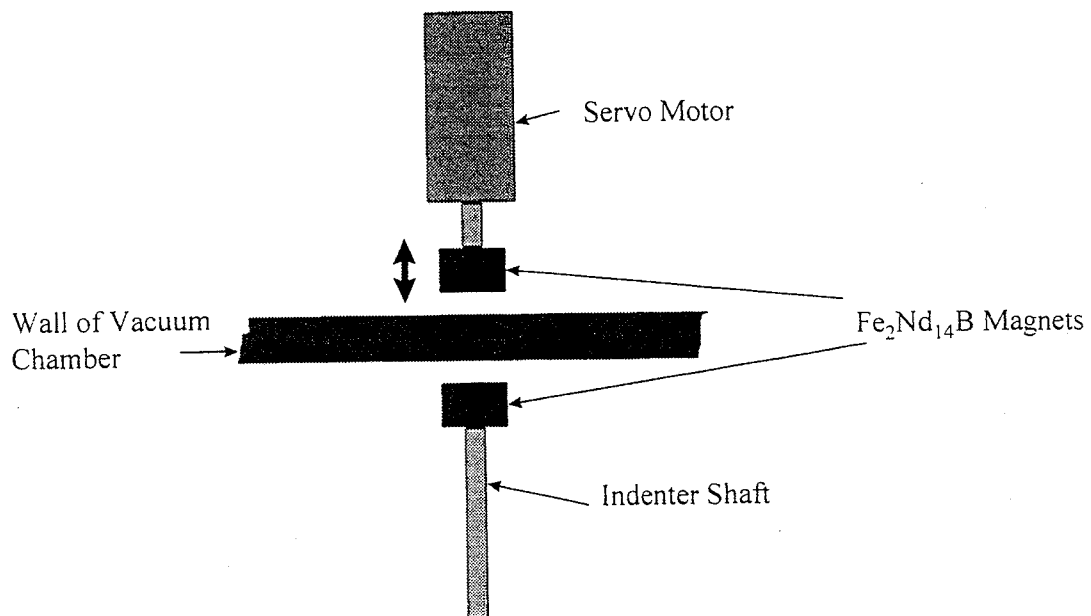


7d Cross section of compression failure (nickel)

Figure 7 Photographs of samples tested using room temperature 4 point bend testing.



Schematic diagram of test arrangement



Schematic diagram of load transfer arrangement

Figure 8 Schematic diagram of hot microhardness apparatus.

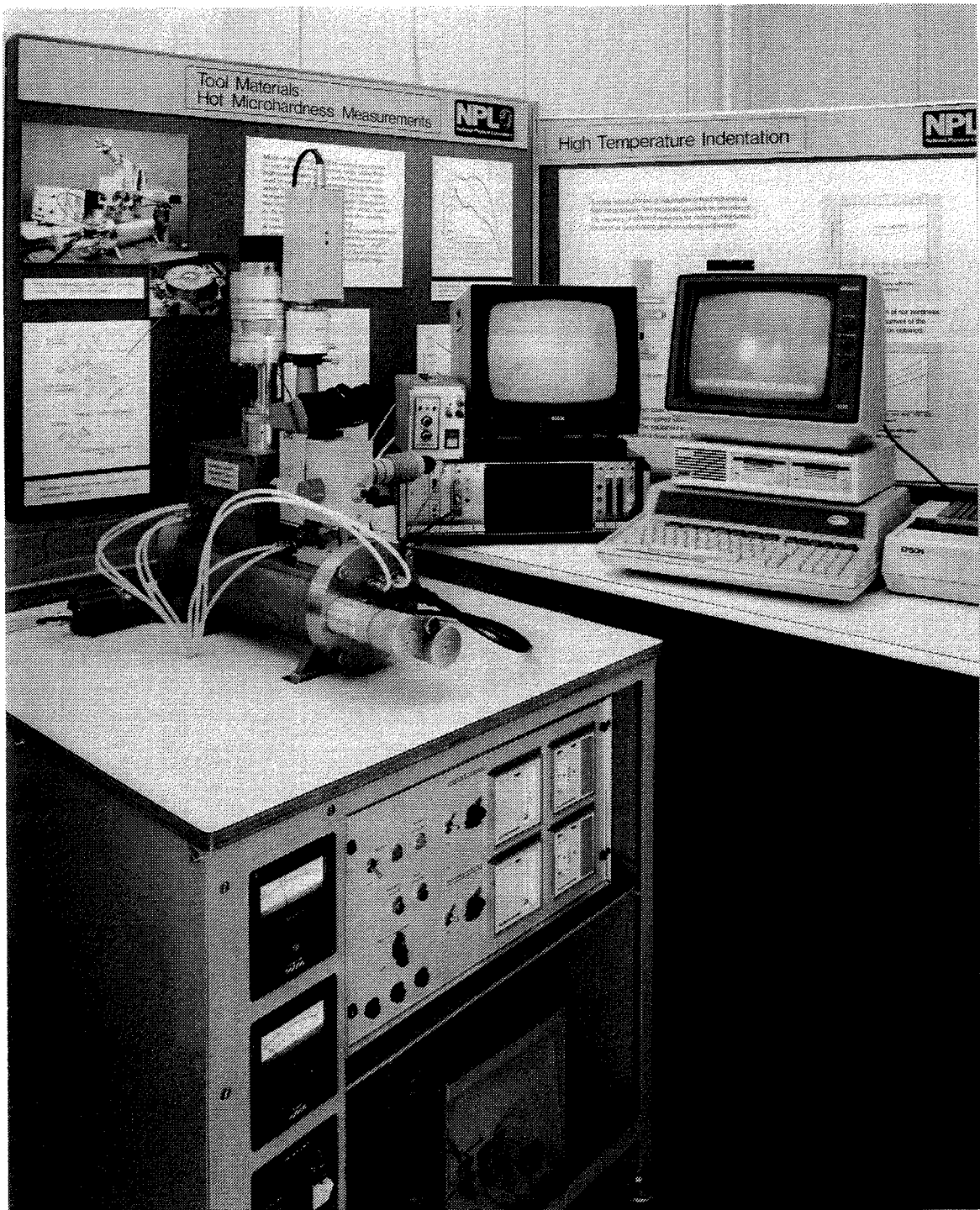


Figure 9 Photograph of hot microhardness apparatus at NPL.

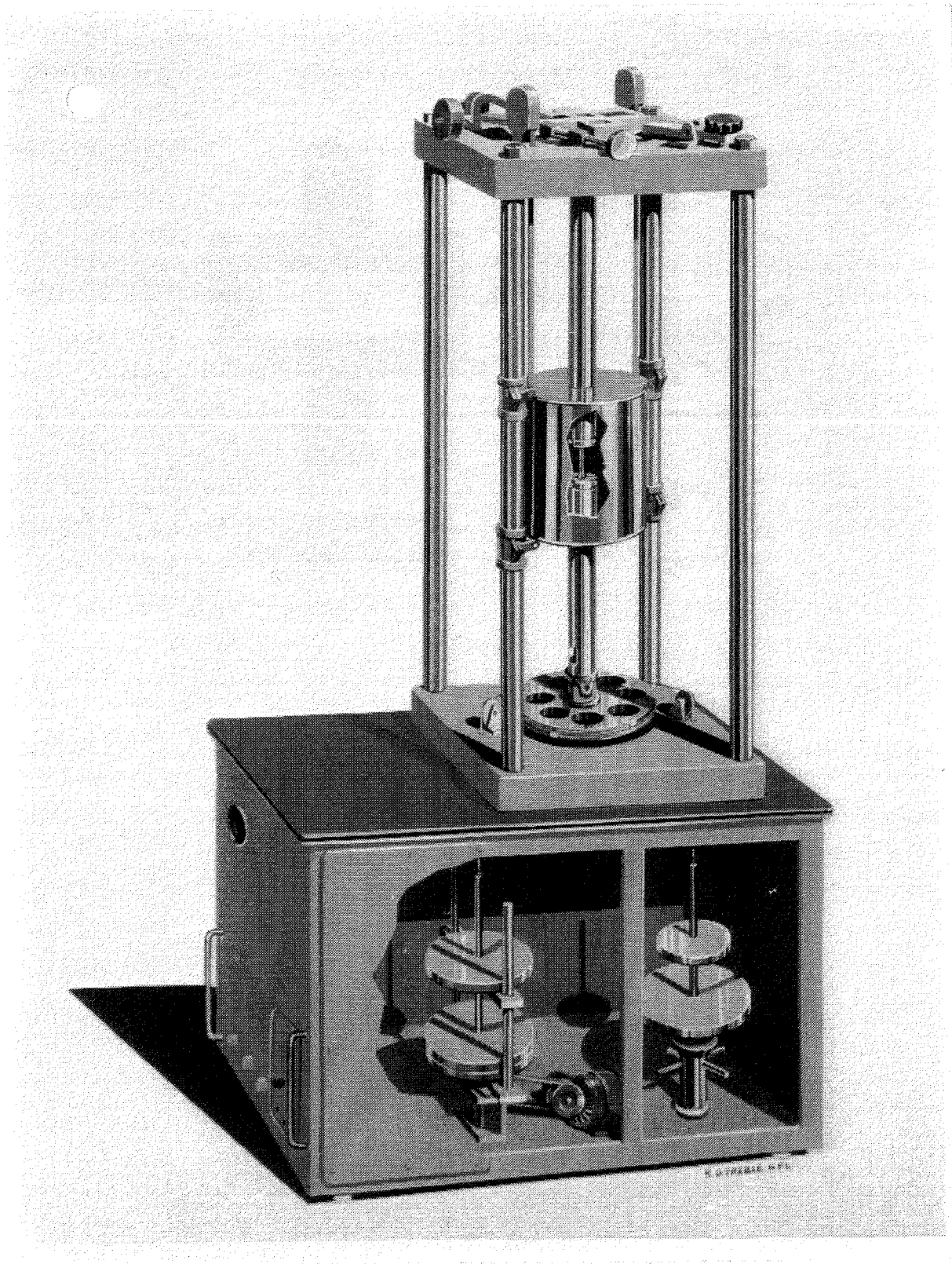


Figure 10 Photograph of NPL designed torsion testing machine.

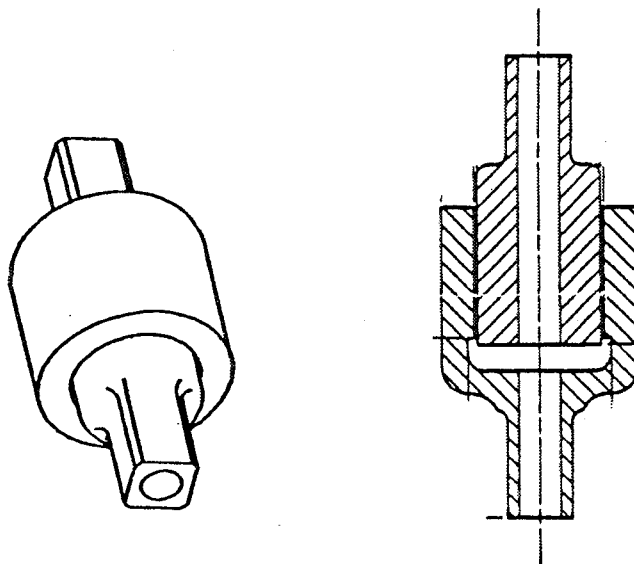
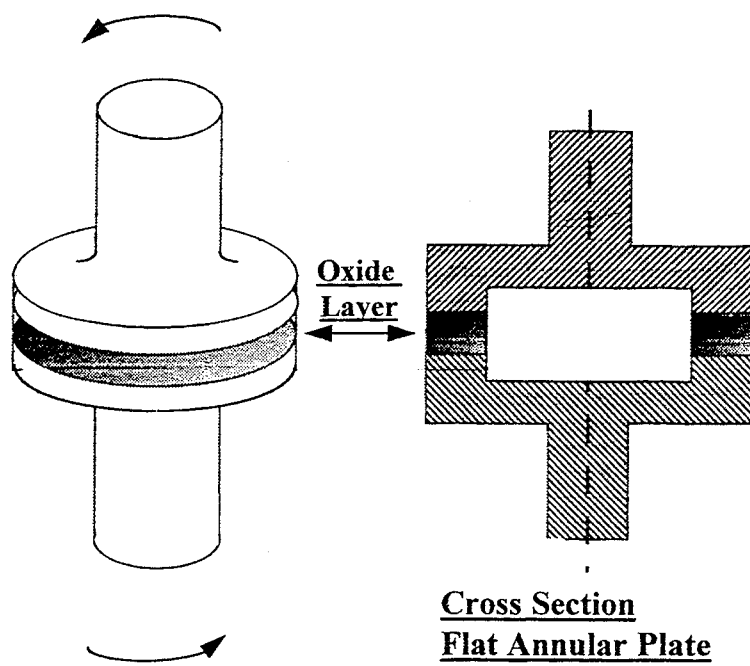


Figure 11 Possible testpiece geometries:

- (a) an annular flat plate
- (b) the spigot/tube testpiece.

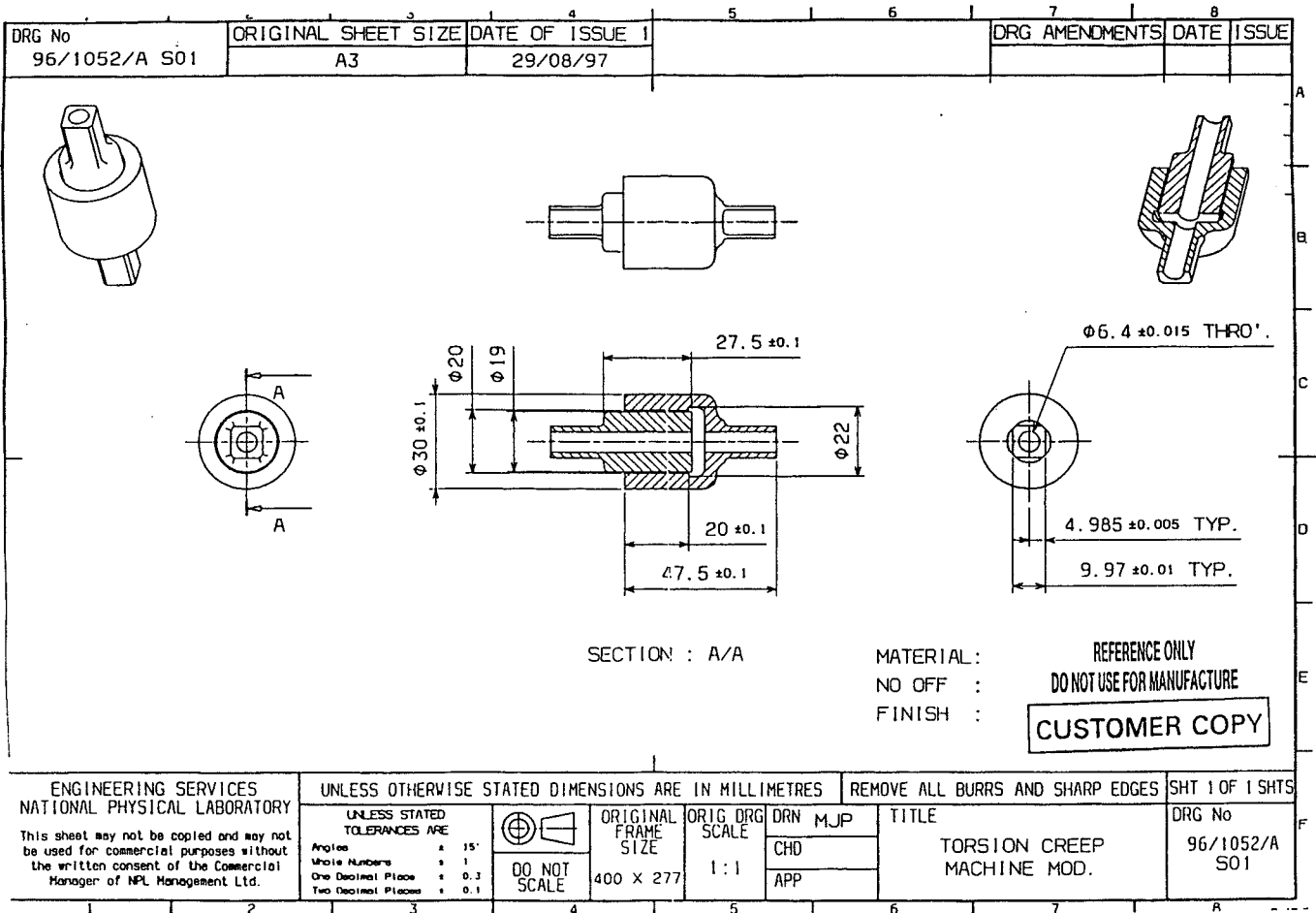
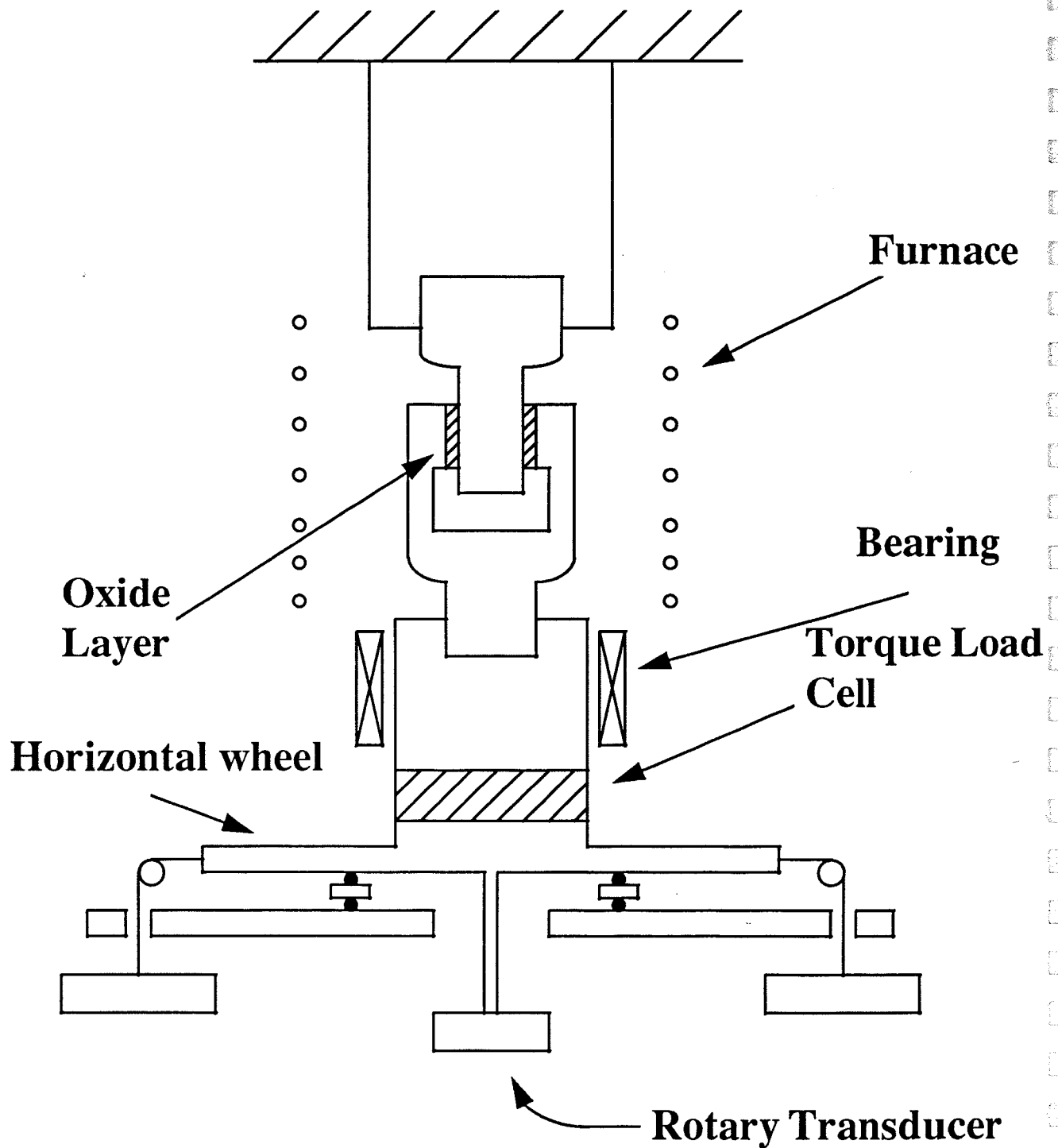


Figure 12 Engineering Services drawing of tube/spigot testpiece.

NPL Torsion Shear Testing Machine.



[Torsion.ppt MSL Euro 06/02/98 Version 1.0]

Figure 13 Schematic design of torsion testing machine for the determination of the strengths of oxides.