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A code of practice for
the use of
Ni-Cr-BASE ALLOY EXTENSOMETERS
FOR MEASUREMENT OF CREEP STRAIN

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HIGH TEMPERATURE MECHANICAL TESTING COMMITTEE

A Code of Practice for the use of
Ni-Cr-BASE ALLOY EXTENSOMETERS FOR MEASUREMENT OF CREEP STRAIN

by

M S Loveday and T B Gibbons

Division of Materials Applications
National Physical Laboratory
Teddington, TW11 0LW.

ABSTRACT

Recommendations for the use of extensometers manufactured from Ni-Cr-base alloys for the measurement of creep strains at high temperature are presented. Procedures for avoiding or minimising the effects of dimensional changes associated with the order/disorder transformation that occurs in these alloys are outlined and technical support for the recommendations is provided in the Appendix. Comparison is made with effects due to dimensional changes resulting from precipitation and from thermal fluctuations.

May 1987

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FOREWORD

This document is one of a series of Codes of Practice issued under the auspices of the High Temperature Mechanical Testing Committee. The aims of the committee, the membership and details of publications are given at the back of this document.

This Code of Practice has been prepared in close consultation with ERA Technology and CEGB and is the result of an initiative of the Advisory Group the 2021 "Creep of Steels" Project at ERA Technology.

INTRODUCTION

The following procedure should be followed when Ni-Cr-base alloys are used for creep extensometry in order to avoid or minimise inaccuracies due to the effects of the order/disorder transformation which occurs at temperatures below about 600 °C in these alloys. It is assumed that the extensometers will be used in accordance with BS 3500[1]. The first four recommendations are generally relevant to any extensometer system and define good testhouse practice; technical support for items 5-12 can be found in the Appendix. The recommendations have taken into account the procedures used at ERA Technology Ltd, and at NPL to minimise measurement inaccuracies in creep testing of steels using extensometers manufactured from Nimonic alloys. This document is based on the information presented in an interim report issue in 1984[2].

RECOMMENDATIONS

1. All limbs of a set of extensometers must be manufactured from the same material.

2. Newly manufactured extensometers should be stress relieved before use by soaking at the service temperature until any changes in dimensions are negligible. [See BS 3846. Clause 5.1.1][3]

3. Individual limbs of a set should be kept together to ensure the same thermal history, and pairs of limbs should always be used together as a matched pair.

4. The individual limbs of a set should be clearly marked with the same primary identifying code, together with individual marks to identify uniquely any particular limb. [Also see 9 below]. Ideally the identifying code should also indicate the material from which the limbs were manufactured if not otherwise marked.

5. For tests below 575 °C, the low contraction alloys should be used for extensometers, see Table 1.

6. If it is impossible to avoid using high contraction alloys for extensometry for service below 575 °C, then it is recommended that for tests at less than 475 °C, the limbs should be soaked at 475 °C

until no further contraction is observed; for tests between 475 °C and 575 °C, the limbs should be soaked at the test temperature until any dimensional changes are negligible.

7. For tests at temperatures above 575 °C extensometers manufactured from precipitation strengthened materials should be pre-annealed at the test temperature to achieve equilibrium transformation products. The time taken for the microstructure to reach a stable condition may range from 100 - 10 000 hours depending upon the material.

8. Extensometers, heat treated to produce the fully ordered state as at (6) above, should be retained only for use at temperatures of less than 475 °C.

9. All sets of high temperature extensometers should be labelled clearly, indicating the temperture range within which they may be used, ie above or below 475 °C.

10. Extensometers manufactured from a high contraction alloy should not be used on successive tests at temperatures above and below the transition temperature.

11. To avoid errors due to differential thermal expansion, the extensometry and the test material should ideally be manufactured from materials having similar coefficients of thermal expansion. This is particularly important for tests involving thermal cycling.

REFERENCES

[1] BS 3500, 1969 (1981). Methods for creep and rupture testing of metals. Pub. British Standards Institution.

[2] M S LOVEDAY and T B GIBBONS "Recommendations for the use of Ni-Cr-base alloy extensometers for measurement of creep strain." NPL Report No DMA(A)81, July 1984.

[3] BS 3846, 1970 (1985). Methods for the calibration and grading of extensometers for testing of metals. Pub. British Standards Institution.

APPENDIX

THE INFLUENCE OF ORDER-DISORDER TRANSFORMATIONS IN Ni-Cr-BASE ALLOYS ON STRAIN MEASURMENT IN CREEP

1 INTRODUCTION

It has been well known for many years that when Ni-Cr-base alloys are cooled through the 400 - 600 °C temperature range a reversible ordering of the crystal lattice occurs which is accompanied by lattice contraction. This can be as much as 0.13% upon long term ageing for certain alloys. More recently it has been recognised that this dimensional instability can give rise to serious inaccuracies in the measurement of creep strains in this range of temperature when these alloys are used in extensometers[1]. The effect was originally noted by ERA Technology Ltd and subsequently by the National Engineering Laboratory and similar types of anomalous strain-time behaviour were observed in other laboratories during the testing of creep resistance steels at approximately 550 °C at low stresses. Instances of anomalously high rates of creep or of negative creep were reported and the behaviour was highly irreproducible. Careful analysis of relevant creep curves, along with x-ray determinations of the contraction in the extensometer material during ordering, provided confirmation that the dimensional instability of the measuring system was responsible for the strain-time behaviour observed[2]. Additionally there is now growing evidence that at temperatures >600 °C dimensional changes occur in the majority of complex engineering alloys due to microstructural precipitation processes which may also result in negative creep[3,4].

The purpose of this note is to summarise the relevance of these effects to the measurement of creep strain and to consider the influence of transformation strains on creep rate and on the shape of creep curves in general.

2 TRANSFORMATION CHARACTERISTICS

2.1 Order/Disorder effects

The kinetics of the order/disorder transformation and the associated dimensional change depend on:

(i) the composition of the material,

(ii) the temperature of the sample in relation to T_c, the order/disorder transition of temperature,

(iii) the time at temperature and

(iv) the thermal pre-history of the material.

Thus when a disordered material is soaked at a temperature substantially below T_c, the ordering reaction will be sluggish and contraction will occur for an extended period of time. However it has been shown for Nimonic 80A that the change from the ordered to the disordered state can be rapid at temperatures just above T_c (Fig 1)[2]. On the basis of this work it can be concluded that this material closely approaches the fully disordered state after annealing for 1h at >575 °C.

Samples of various materials commonly used for high temperature extensometers aged at NPL at 475 °C for times up to 20 000 h were measured for lattice parameter contractions by B Nath of CERL, UK and A Marucco of ITM, Italy, and a summary of their findings is given in Table 1. It can be seen that the range of alloys investigated may be classified in two groups, viz low contraction alloys exhibiting lattice contractions of less than 0.025% after 10 000 h ageing, and high contraction alloys showing contractions of greater than approximately 0.035% for a similar ageing history. Several of the latter group exhibited contractions as large as 0.1% after prolonged ageing. Also listed is the suggested usable range of the extensometer materials.

2.2 Precipitation effects

The majority of the complex nickel base superalloys are strengthened by the precipitation of γ'. Both γ' and the various carbides coarsen with exposure at elevated temperature and volumetric contraction and changes in density may result. The precise amount of contraction depends on the composition of the alloy and the exposure time at elevated temperature. Typical contractions of approximately 0.05% have been reported at temperatures between 600 °C - 1000 °C after exposure times of 100 - 5 000 hours, and values of negative creep as large as 0.1% have been reported for Waspalloy at 800 °C.[3] There is also limited evidence for certain alloys indicating that after an initial contraction the material subsequently expands, particularly at higher temperatures.

3 IMPLICATIONS FOR MEASUREMENT OF CREEP STRAIN

Although several different configurations of extensometer - transducer arrangement can be used (see for example Fig 2) each will be sensitive to the effects of order/disorder changes in the material. This applies even in instances where the creep test temperature is significantly above T_c since at some stage, part of the extensometer limb will pass through the critical temperature range. It is therefore essential for good measurement practice to ensure that the limbs of extensometers are always kept together in pairs, or in the case of side-to-side averaging extensometers, all four limbs are kept together as a complete set. This ensures that thermal history is not a variable.

Thus, provided appropriate steps are taken and careful records of extensometer usage are maintained, it should be possible to minimise the effects of dimensional instability on strain measurement. Certain situatons, however, could give rise to large errors and should be avoided. These are:

(a) the use of extensometer limbs in the fully disordered state when the test temperature is in the range where ordering occurs;

(b) the use of extensometers at test temperatures above T_c which had previously been used at a temperature where ordering occurred.

In the UK most creep test laboratories aim to comply with the conditions laid down in BS 3500 which in turn specifies that extensometry should be calibrated in accordance with BS 3846. In the latter standard it is recommended that extensometers should be calibrated at the test temperature, (clause 5.1.1). However in practice calibration is normally carried out at room temperature. BS 3846 therefore recommends procedures should be adopted to ensure that negligible drift of the strain reading occurs with time and hence the dimensional stability of new extensometers should be checked by measuring any dimensional changes during prolonged soaking at temperatures somewhat higher than those to be used in practice. This topic has been discussed in some detail by Day and Harrison[5].

In Fig 2 various creep curves are schematically illustrated showing the dependence of curve shape on both transducer configuration and the relationship of test temperature to T_c, the order/disorder transition temperature. If the rate of accumulation of creep strain is equal in magnitude but in the opposite direction to strain associated with an order/disorder transformation, then the resultant creep curve will exhibit a plateau where the creep rate is zero, which may be mistakenly interpreted as some form of incubation period.

It should be pointed out that similar shapes of creep curve would be produced as a result of the order/disorder transformation in a testpiece when strain was measured using non-ordering extensometers.

4 EFFECT ON CREEP RATE

When primary creep strains are large any dimensional instability of the extensometer will be insignificant but, at temperatures such that the ordering or precipitation kinetics are slow, the effect of dimensional changes may become apparent in the secondary creep stage as indicated schematically in Fig 2. Assuming that the maximum change in dimensions due to the order/disorder transformation is 0.13%[6] then the time taken for the creep strain at a given creep rate to exceed the strain induced by order/discorder effects can be estimated from Fig 3. The time taken for the transition to be completed depends on the test temperature and on the kinetics of the process, but it can be seen from Fig 3 that at a creep rate of 10^{-5}/h, which is relatively fast for power engineering steels, 130 hours would elapse before the creep strain exceeded the strain due to the order/disorder transition. Thereafter the creep strain will always be larger and the true shape of the creep curve will become apparent. However, it should be noted that the order/disorder strain will still contribute at least a 10 per cent error in creep strain for times up to an order of magnitude after that at which the two strains are equal. A similar argument holds true if dimensional changes are due to precipitation or instability of transformation products at higher temperatures.

5 COMPARISON WITH STRAINS DUE TO THERMAL FLUCTUATIONS

BS 3500 specifies the maximum allowable fluctuation in test temperature is ± 3 °C for tests of more than 100 h duration at temperatures up to 600 °C duration and ± 4 °C for tests between 600 °C - 800 °C. Thus the maximum temperature excursions allowed are 6 °C and 8 °C respectively. The maximum effects will occur in situations where Nimonic alloy extensometers are being used for strain measurement for alloys, such as austenitic steels, with a significant difference in coefficient of thermal expansion ($3.8 \times 10^{-6} K^{-1}$). The strains due to differential thermal expansion in these conditions are shown in Fig 3 and it is clear that any errors due to this effect will be small compared with those associated with the order/disorder transformation.

6 ACKNOWLEDGEMENT

There have been helpful discussions with Mr A Wickens, formerly of ERA Technology, Drs E Metcalfe and B Nath, CERL and Dr B F Dyson, NPL.

7 REFERENCES

[1] P F APLIN "Minimisation of errors due to metallurgical instability in high nickel alloy extensometry: A case history" High Temperature Strain Measurement, Ed. R C Hurst et al, Elsevier Applied Science, London 1986.

[2] A MARUCCO, E METCALFE and B NATH "The order/disorder transformation in Ni-Cr alloys". Int. Conf. Solid-Solid Phase Transformations. Pittsburgh, August 1982.

[3] B REPPICH "Negative Creep" Z. fur Metallk., 75, No 3, 193-202, 1984.

[4] R TIMMINS, G W GREENWOOD and B F DYSON "Negative creep in a nickel-base superalloy", Scripta Met, 20, 67-70, 1986.

[5] M F DAY and G F HARRISON "Design and calibration of extensometers and transducers", Measurement of high temperature properties of materials, M S Loveday, M F Day and B F Dyson Eds, p.225. London HMSO 1982.

[6] B NATH and E METCALFE Private communication. 1986.

MATERIAL	ORDER/DISORDER STRAIN after 10 000h at 475 °C %	after 20 000h at 475 °C %	Suggested normal usable extensometer range °C
Low Contraction Alloys			
20 Cr-25Ni stainless steel	0.024*	-	RT - 575
316 stainless	0.025	-	RT - 575
ALLOY 600 (Sanicro 71)	0.022*	-	RT - 750
INCONEL X750	0.022	-	RT - 800
High Contraction Alloys			
NIMONIC 80A	0.035	0.107 0.13**	575 - 825
NIMONIC 90	0.042	0.073	575 - 875
NIMONIC 100	0.036	0.045	575 - 875
NIMONIC 105	0.068	0.086	575 - 950
NIMONIC 115	0.079	0.102	575 - 1000

* Murucco and Nath, unpublished data

** Aged for 70 000 hours

NOTE The cast alloys IN713, IN100 and Mar M002 are also used for extensometry and loading bars for temperatures up to about 1100 °C. Measurements of lattice concentration were made for IN713 and Mar M002 but no satisfactory results were obtained.

TABLE 1 - Materials used for creep extensometers and loading bars

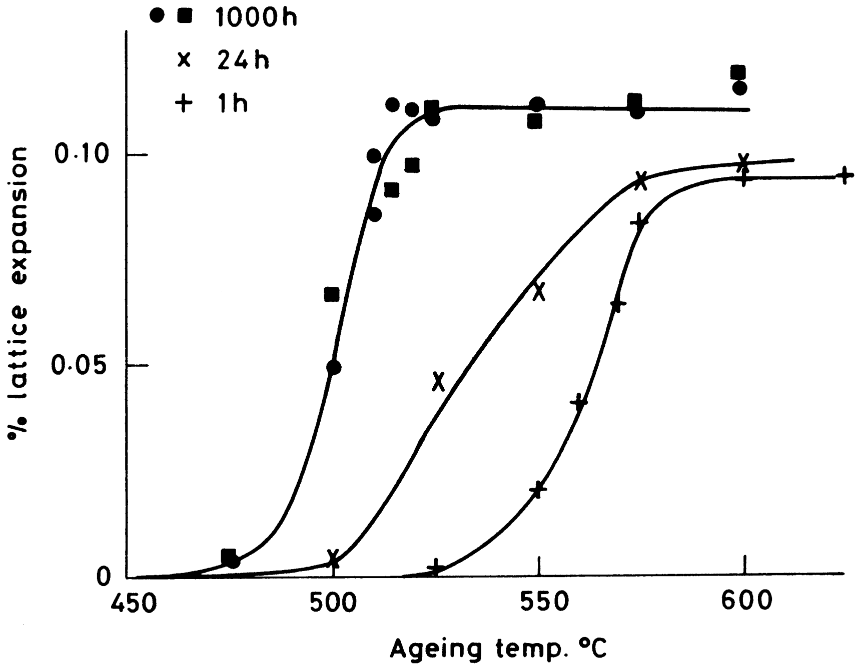

Fig 1 Lattice expansion of preordered Nimonic 80A upon heating. Initial condition: 450 °C, 30 000h without stress (+ x ●) and under stress (■) [Ref 1]

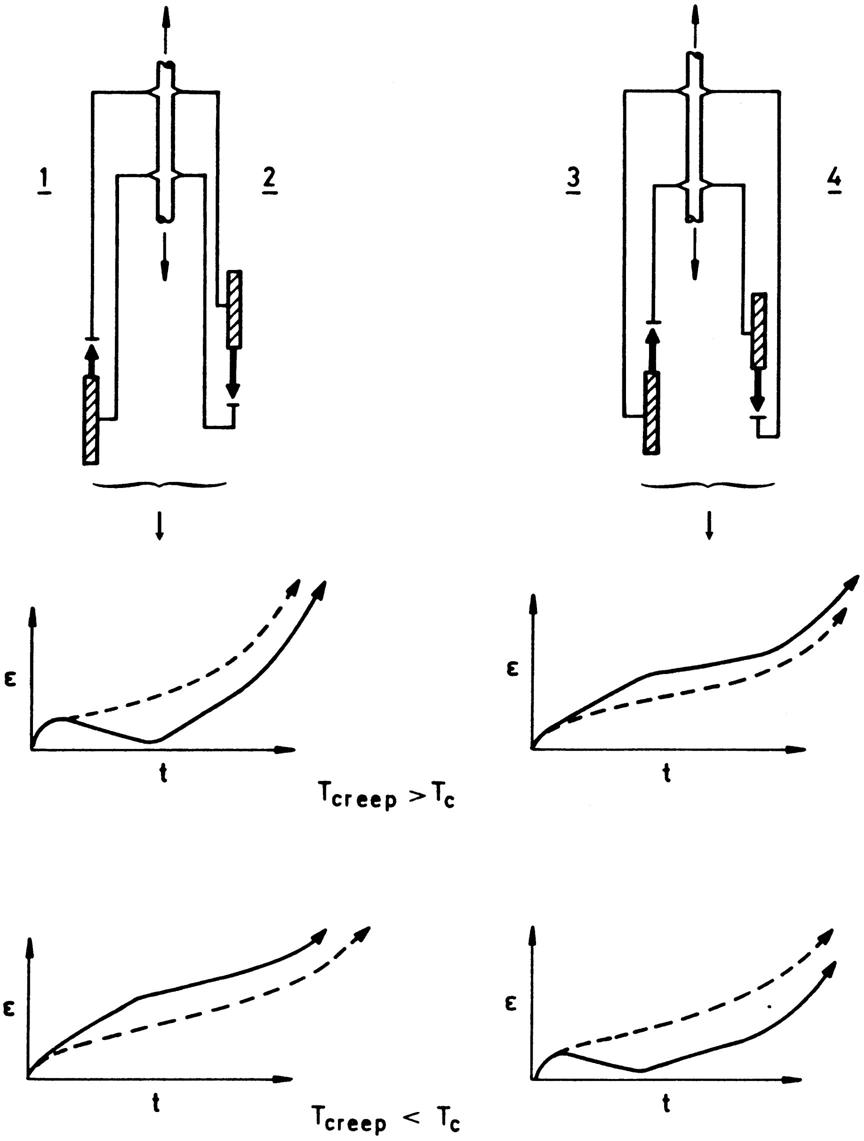

Fig 2 Schematic diagram showing 4 possible classes of extensometer - transducer configuration, and the resulting creep curves obained depending upon whether the creep test temperature is above or below the order/disorder transformation temperature, T_c. The upper curves apply for extensometers initially in the ordered condition, whilst the lower curves correspond to extensometers initially in the disordered condition, ie solution annealed. It is assumed that the test piece is a non-ordering material.

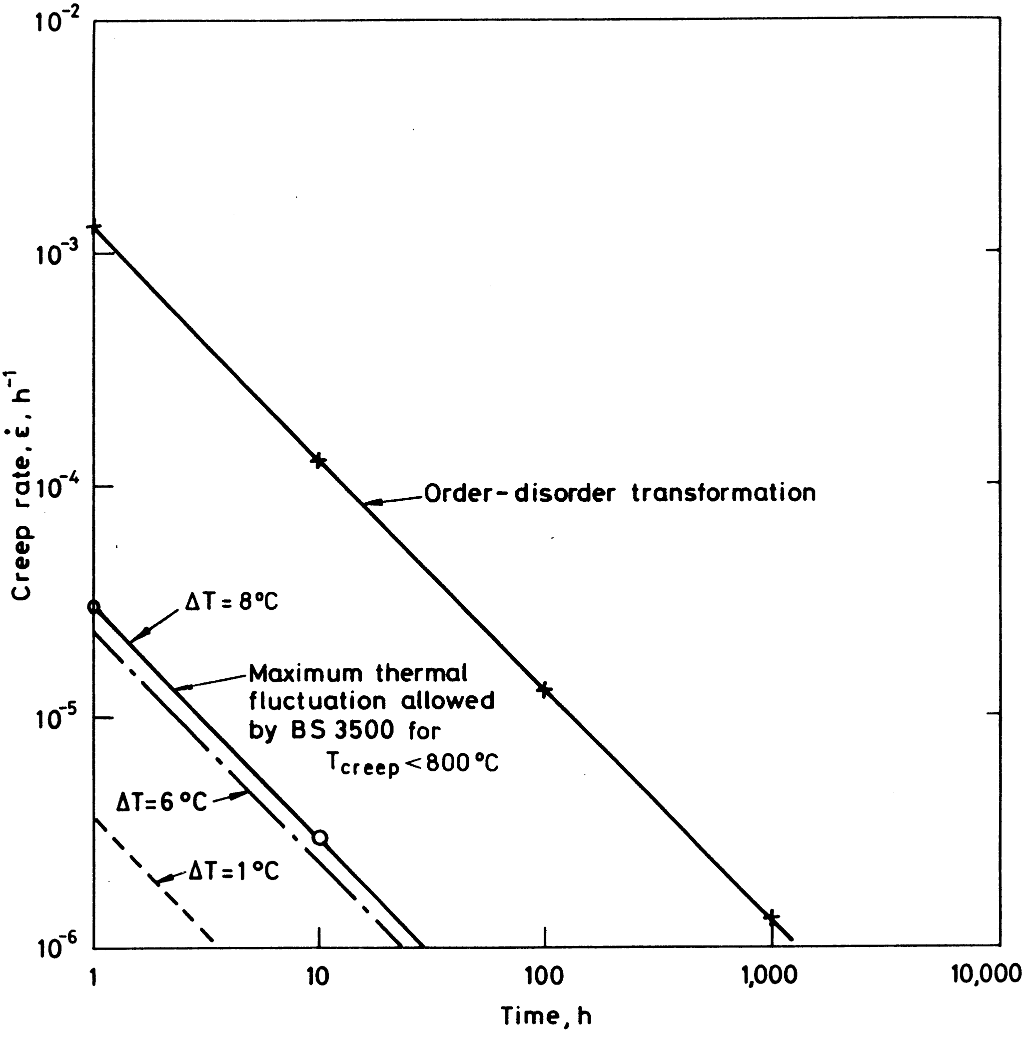

Fig 3 Creep rates due to strains associated with thermal fluctuations and with the order/disorder transformation as a function of time.

PUBLICATIONS

Books

"Measurement of High Temperature Mechanical Properties of Materials"

Ed. M S Loveday, M F Day and B F Dyson
Pub. HMSO London (1982)
(ISBN 0 11 480049 9)

Hardback, 20 chapters, 350 pages
Price: £30

Topics covered include testing standards, temperature measurement, load-cell calibration, design of creep and fatigue machines, extensometer design, measurement of crack growth, thermal fatigue, environmental testing, specimen design etc.

This book has become recognised as a standard manual on high temperature testing techniques and provides a useful source of references to original research papers on the subject.

"Techniques for High Temperature Fatigue Testing"

Ed. G Sumner and V B Livesey
Pub. Elsevier Applied Science (1985)
(ISBN 0 85334 314 4)

Hardback, 10 chapters, 200 pages
Price: £26

Proceedings of a conference held in Preston in 1984, organised in conjunction with UKAEA Springfield. The book contains valuable information on the design of high temeprature fatigue testing machines and the measurement and control of testing parameters.

"Techniques for Multi-axial Creep Testing"

Ed. D J Gooch and I M How
Pub. Elsevier Applied Science (1986)
(ISBN 1-85166-033-X)

Hardback, 19 chapters, 360 pages
Price: £40

Proceedings of a conference held at Leatherhead in 1985, organised jointly by CERL and ERA Technology Ltd. This book contains important information relating to all aspects of high temperature multi-axial creep testing.

Code of Practice

"A Code of Practice for **Constant-Amplitude Low Cycle Fatigue Testing** at elevated temperature", by G B Thomas, R Hales, J Ramsdale, R W Suhr and G Sumner. (Low Cycle Fatigue Working Party). Pub. NPL 1986
(ISBN 0-946754-05-5)

Softback, 43 pages.
(Available free on application to M S Loveday, NPL).

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The committee was established in 1982 following a conference held at NPL on 'Measurement of High Temperature Mechanical Properties of Materials'. The membership of the committee includes experts in the field of high temperature testing drawn from industry, research institutions and universities.

For further information contact:

Malcolm S. Loveday
HTMT Committee Secretary
Division of Materials Applications
National Physical Laboratory
Teddington, TW11 OLW

Phone: 01-977 3222
Telex: 262344