PHILOSOPHICAL TRANSACTIONS OF THE ROYAL SOCIETY A MATHEMATICAL, PHYSICAL & ENGINEERING SCIENCES

Mechanical Testing of High-Temperature Materials: Modelling Data-Scatter [and Discussion]

B. F. Dyson, P. Hirsch, M. McLean, R. C. Thomson and M. S. Loveday

Phil. Trans. R. Soc. Lond. A 1995 **351**, 579-594
doi: 10.1098/rsta.1995.0054

Mechanical testing of high-temperature materials: modelling data-scatter

By B. F. Dyson
Department of Materials, Imperial College of Science, Technology and Medicine, London SW7 2BP, UK

Scatter in mechanical property data of a metallic alloy derives from two sources: a variable microstructure or an inadequate and sometimes badly executed test method. A quantified example of each source of scatter is given in this paper by examining two important high-temperature properties: uniaxial tensile creep and low-cycle fatigue. Creep-lifetime data of engineering alloys often show scatter-bands much greater than can be accounted for on the basis of well-understood physics of testing-induced scatter. Bounds to this material-induced scatter have been computed for a low-alloy ferritic steel dataset using a physically based creep model incorporating damage state variables. High-temperature low-cycle fatigue demands a more complicated test procedure than does steady-load creep and the large interlaboratory scatter found in recent round robin data from 26 laboratories in Europe and Japan has highlighted inadequacies in the standardized test method. A simple material model and fracture criterion has been used, in conjunction with a previously introduced testpiece-bending model, to predict testing-induced interlaboratory scatter for the two nickel-base superalloys reported upon in the round robin exercise.

1. Introduction

Low-cycle fatigue and creep are two of the most important high-temperature mechanical properties in aero-engine applications and their laboratory-testpiece data often show a large scatter. Mechanical property data-scatter is universal and its degree depends on both the material and the testing procedures. Material-induced scatter is caused by gross spatial variability in the material's microstructure within a single 'cast' or to microstructural variability between different casts: the 'tightness' of the chemical specification; the shape and size of the component from which the testpiece is manufactured; and the component's thermo-mechanical processing route all contribute towards the extent of microstructural inhomogeneity. It is only through qualitative (or preferably quantitative) implementation within the processing technology of expensively acquired microstructural and kinetic knowledge that some control can be exerted on the magnitude of this cause of data-scatter. The more mature a material is in its engineering usage, the more likely there will be an adequate knowledge-base to facilitate close microstructural control. Whether implementation will be economically worthwhile is another matter; the specific product will certainly need to have sufficient added-value, which goes some way towards understanding why

Phil. Trans. R. Soc. Lond. A (1995) **351**, 579–594
Printed in Great Britain

microstructural control has progressed more in aero-engine applications than in, for example, petrochemical plant.

Data-scatter resulting from inadequacies in creep and low-cycle fatigue (LCF) testing has three main constituents: (i) random and systematic errors in measurements made during testing; (ii) random fluctuations in the magnitudes of the test control-parameters: force/stress, displacement/strain and temperature; (iii) errors in the computation of strain due to uncertainties in the magnitude of the operative gauge length and/or its local state of loading. Testing-scatter has received a high profile over the last decade as a consequence of the drive towards harmonization and accreditation of testing methods, aimed at removing potential barriers to trade in the global market. Research has been sponsored in Europe by the Community Bureau of Reference (BCR) and fostered internationally by the Versailles Project on Advanced Materials and Standards (VAMAS). *Repeatability* of a dataset within a single laboratory and *reproducibility* of the equivalent dataset between laboratories rely in the first instance, on the existence of Measurement Standards for length, mass, time, temperature, etc., and secondly on Documentary Standards. The latter contain not only methodologies for performing what are often highly complicated materials property tests, but provide a framework for *documented* traceability to Measurement Standards (Dyson *et al.* 1995; Hossain & Sced 1995).

Lack of precision in data is not always an issue when materials are being considered for structural applications – for example in the early stages of design (Ashby 1989, 1991) – but once a material class has been chosen for a particular application, data-scatter poses an obvious problem for a design engineer hoping to steal a competitive-edge in a global market place that is increasingly unforgiving of failure to deliver on time and to specification. In Europe, the harmonization process may itself cause an increase in data-scatter in the future, as a consequence of its 'lowest common denominator' policy towards test methods (Loveday 1992). This is ironic considering that the potential of cheap computer-based design and lifetime prediction will become more difficult to realise if the input data (condensed though it will be, as constitutive laws) is insufficiently precise.

Since data-scatter is a fact of life, its physical origins require quantitative understanding if full use is to be made of materials data in structural design, although this is not the route currently chosen by European Standards committees, which take a purely statistical stance, probably due to the fact that physically based methods of quantifying data-scatter are only now emerging. This paper focuses on two recent advances in predictive modelling of data-scatter: in creep, when the source of scatter lies primarily in 'cast-to-cast' microstructural differences; and in low-cycle fatigue, when scatter can be attributed to inadequacies in the test method.

2. Modelling materials-scatter in creep

(a) Background

Within an advanced and frequently accredited (Henderson & Thomas 1995) creep-testing laboratory, random and systematic errors in basic metrology should be small and contribute little to data-scatter: the resolutions of devices for measuring displacement, force and temperature, recommended in Documentary Standards, are usually more than adequate for the task. An exception being the de-

PHILOSOPHICAL TRANSACTIONS OF THE ROYAL SOCIETY A MATHEMATICAL, PHYSICAL & ENGINEERING SCIENCES

termination of Young's modulus using testpieces with a short (but still within standard) gauge length (Loveday 1992). Similarly, fluctuations in the control parameters (the so-called tolerances) can, using modern devices, be easily kept within the ranges allowed by Documentary Standards. In uniaxial creep testing (which accounts for the majority of creep strain data), there are systematic errors generated during the computation of strain from displacements made on testpieces having extensometer ridges, because the accompanying non-uniform strain fields give rise to an uncertain gauge length. Extensometer ridges are used world-wide in creep testing and a comprehensive finite element study has recently been published of the errors in strain that are predicted to occur when superalloy behaviour is simulated using a multiaxial material model with tertiary-damage variables (Lin *et al.* 1993*a*). Testpieces with nominal gauge lengths greater than approximately 40 mm – universally used to generate so-called high-sensitivity creep strain data – are predicted to have only small errors from this source and these can be reduced to negligible levels using a modified design for the ridge (Lin *et al.* 1993*b*).

Uniaxial creep data should therefore be procurable with a minimum of reproducibility errors caused by inadequacies in testing procedures. This conclusion is further justified by the relatively recent validation of a Creep Reference Material (Gould & Loveday 1992), where it was demonstrated that scatter in strain/time trajectories exhibited by the Reference Material could be accounted for by the tolerances on stress and (particularly) temperature specified in Documentary Standards. Figure 1 illustrates this with a set of six creep curves generated by a single laboratory and reported by Gould & Loveday (1992): the scatter markers shown in figure 1 were calculated from the tolerances specified in BS 3500 and ISO DIS783 using a simple power-law creep model with exponential temperature dependency. The sigmoidal primary creep displayed in figure 1 is unusual for this material (a specially prepared batch of Nimonic 75) but is a useful monitor of testing proficiency.

In spite of an adequate test procedure, scatter in creep lifetime data of engineering alloys is widespread and particularly bad in the extensive databases that have been generated for ferritic steels used as high-temperature structural components in electricity power plant. It is unlikely that scatter is caused by poor implementation of the creep test procedure and indeed the electricity supply industry devoted much effort in the 1960s establishing this point. Figure 2 is the only comprehensive systematic study known to the author that clearly and unambiguously relates thermally induced microstructual changes in an important engineering material with subsequent scatter in creep lifetimes. Toft & Marsden (1961) classified the microstructures of 1%Cr1/2%Mo steel tube components that had been exposed to a variety of thermal environments in service before laboratory creep testing; each symbol in figure 2 represents a particular microstructure, although in practice there was necessarily some subjectivity in the classification. Toft & Marsden made the very interesting point that individual extrapolation of each dataset using the empirical Manson-Haferd (1953) method predicts a common lifetime of 10^5 h for all microstructures at a stress of approximately 30 MPa. It is suggested here that the connection between Toft & Marsden's work and the scatter exhibited by ferritic steel creep data in general lies in the fact that the processing route for these materials naturally results in a spectrum of microstructures between casts and that this is the underlying cause

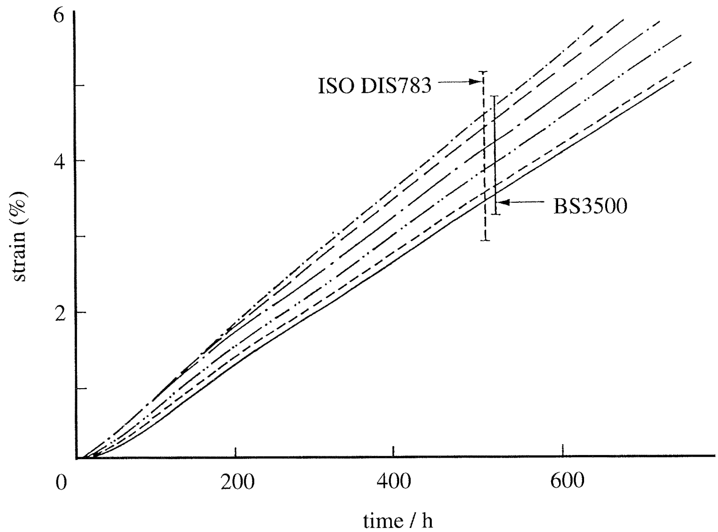

Figure 1. Illustrating the adequacy of uniaxial tensile creep testing practice using a Nimonic 75 Creep Reference Material (Gould & Loveday 1992).

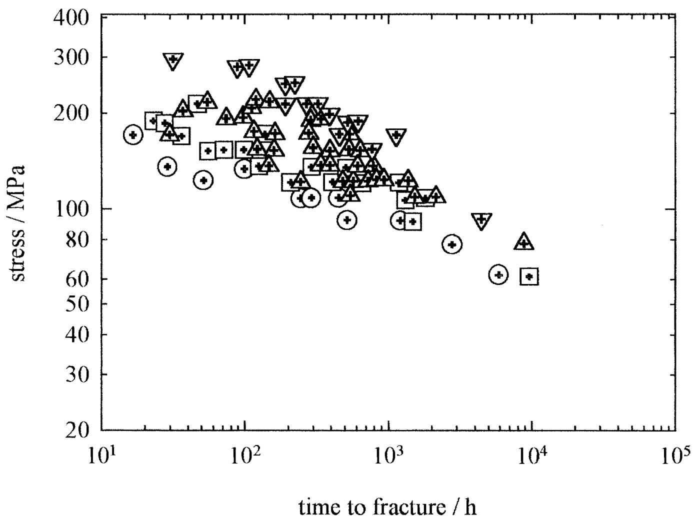

Figure 2. Time to fracture as a function of stress for four different batches of 1%Cr1/2%Mo steel (replotted data of Toft & Marsden 1961).

of the data-scatter. The working hypothesis in this paper is that the primary detailed micromechanism of material-scatter in ferritic steels lies in the volume fraction and dispersion characteristics of the main carbide-strengthening particles and that efficient extrapolation procedures will only emerge through quantitative creep modelling.

(*b*) *Material model*

The uniaxial creep behaviour of precipitation hardened alloys has recently been modelled by Dyson & Osgerby (1993). Creep is hypothesized as being rate-limited by the drift of dislocations surmounting a spherical particle distribution which

Table 1. *Model material creep parameters for a* $1\%\mathrm{Cr}\frac{1}{2}\%\mathrm{Mo}$ *ferritic steel*

$\dot{\epsilon}_0/\mathrm{h}^{-1}$	σ_0/MPa	h'/MPa	H^*	K'/h^{-1}
$1.4\times10^9\sigma_0^{-1}\mathrm{e}^{-31\,000/T}$	$8\times10^{-3}\mathrm{e}^{6000/T}$	10^5	0.4	$1.4\times10^{12}\sigma_0^3\mathrm{e}^{-36\,000/T}$

is spatially homogeneous. Drift occurs by climb and glide occurring in parallel along any dislocation segment and leads to an activation-area that reflects the state of precipitate dispersion, thus predicting an effect of coarsening and/or volume fraction which manifests itself either as tertiary creep or as an initial weakening when the material has been given a prior thermal exposure. Creep behaviour is given by the constitutive and damage evolution equation-set:

$$\left.\begin{aligned} \dot{\epsilon} &= \dot{\epsilon}_0 \sinh \frac{\sigma(1-H)}{\sigma_0(1-S)(1-\omega)}, \\ \dot{H} &= h'(1-H/H^*)\dot{\epsilon}/\sigma, \\ \dot{S} &= \tfrac{1}{3}K'(1-S)^4, \\ \dot{\omega} &= D\dot{\epsilon}. \end{aligned}\right\} \tag{2.1}$$

Equation-set (2.1) differs in two ways from previous attempts (reviewed by Dyson & McLean 1990; McLean *et al.* 1991) to provide physically based constitutive and damage evolution equations for predicting creep behaviour: the new creep mechanism has a sinh stress-function, rather than the more familiar power-law and thereby models minimum creep rates well; the effect of particle coarsening enters the strain rate equation as a stress multiplier, rather than as the usual threshold stress.

The first term in equation-set (2.1) defines the true creep rate, $\dot{\epsilon}$, in terms of (i) the uniaxial stress, σ; (ii) the hardening parameter, H, causing primary creep due to internal stress redistribution; (iii) the softening parameter, S, causing tertiary creep by the Lifshitz–Slyozov (1961) particle-coarsening equation; and (iv) the softening parameter, ω, causing tertiary creep and fracture. The unusual form of the Lifshitz–Slyozov (1961) equation is a consequence of the parameter S being defined by $S = 1 - p_i/p$ to range between zero and unity; p is the interparticle spacing and the subscript denotes an initial value. Explicit expressions for $\dot{\epsilon}_0$ and σ_0, given by Dyson & Osgerby (1993), are not sufficiently accurate for lifetime prediction and were treated, along with h', K' and H^*, as model parameters to be determined from creep data analysis, but constrained by physics to lie within certain ranges. Table 1 gives the parameters derived by Dyson & Osgerby, from 1%Cr1/2%Mo steel data generated in a COST 501 programme of the European Community.

The activation energy in $\dot{\epsilon}_0$ is nominally that of self-diffusion, while that for K' additionally contains the heat of solution of carbon in iron. The COST programme used material that had been either newly processed or service-exposed for 250 000 h at 525 °C; table 1 applies to new material and to material service-exposed at temperatures greater than 883 K. For material service-exposed at temperatures greater than 800 K but less than 883 K, $|\sigma_0|$ is replaced by the

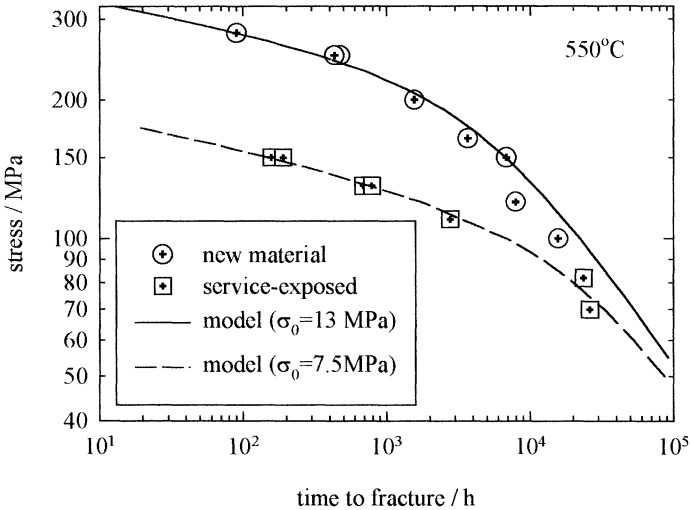

Figure 3. Illustrating the ability of equation-set (2.1) to model the substantial differences in fracture lifetimes between new 1%Cr1/2%Mo steel and serviced-exposed material. The only model parameter to be changed is σ_0, which quantifies the coarsening of the particles.

following:

$$\sigma_0 = 1.62 \exp[1300/T] \text{ MPa}. \tag{2.2}$$

Equation (2.2) reflects the effect of thermal ageing during service exposure on the initial carbide particle spacing, while the activation energy is believed to be related to the temperature dependence of the particulate volume fraction. Table 1 and equation (2.2) have been used as input parameters in equation-set (2.1) to generate the solid and dashed curves in figure 3 which, in qualitative agreement with the empirical calculations of Toft & Marsden (1961), predict convergence of behaviour in new and service-exposed material at times of the order of 10^5 h.

(c) *Predicted creep data-scatter*

Figure 4 compares the lifetime/stress data of Toft & Marsden (1961) with the behaviour predicted using the COST model material parameters given in table 1, from which a value of $\sigma_0 = 10.5$ MPa can be calculated at a temperature of 565 °C. The predicted behaviour lies not only within but, more importantly, towards the top of the scatter band (consistent with the Toft & Marsden data being service-exposed) and so gives confidence in the underlying physics of the model. It's potential usefulness in extrapolating data that are scattered primarily because of large-scale material inhomegeneities is illustrated in figure 5: the two solid lines bounding the data were calculated using values of $\dot{\epsilon}_0$, K', h' and H^* given in table 1 calculated at 565 °C, with two values of σ_0 to reflect the extremes of microstructure.

3. Modelling testing-scatter in low-cycle fatigue

(a) *Background*

In 1985, an intercomparison programme on high-temperature low-cycle fatigue (LCF) was sponsored by BCR, with the UK's National Physical Laboratory (NPL)

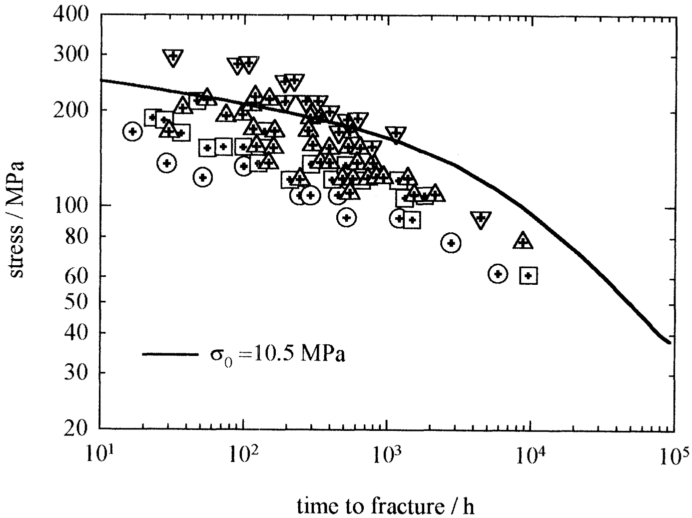

Figure 4. Comparison of Toft & Marsden (1961) data with predictions using equation-set (2.1) and COST model parameters.

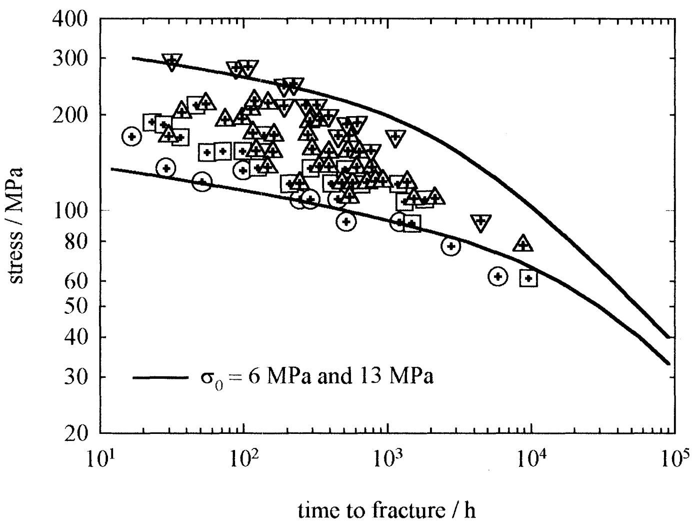

Figure 5. Bounding of Toft & Marsden (1961) data using equation-set (2.1) and COST model parameters with two values of σ_0.

taking the lead. The proposal had evolved from an initiative by the UK's High Temperature Mechanical Testing Committee to prepare a code of testing practice (Thomas *et al.* 1989), based upon an emerging European consensus on best practice. Four types of metallic alloy were tested: AISI 316L steel, 9Cr1Mo steel and IN 718 at 550 °C; and Nimonic 101 at 850 °C. Twenty-six laboratories in Europe and Japan produced 61 sets of LCF data which were subsequently analysed by Thomas & Varma (1992). Laboratories were requested to test three testpieces at each of 3 prescribed strain ranges for every alloy: in the event, not all laboratories tested every alloy and some used more than one type of extensometer in order to explore their differences. The results within any one laboratory showed little

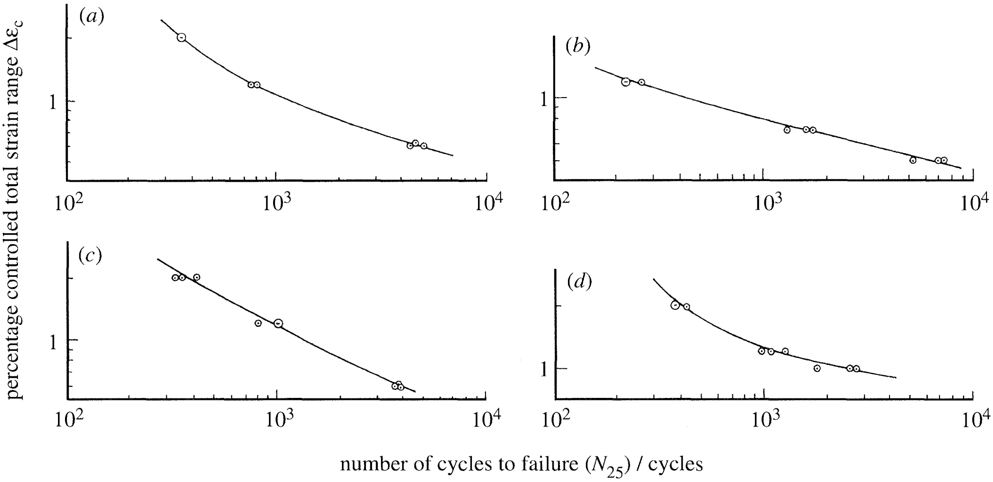

Figure 6. Four sets of low-cycle fatigue test results from a single laboratory in the BCR round robin exercise (original data from Thomas & Varma 1992).

scatter and examples of this good repeatability are given in figure 6. In contrast, reproducibility of data between laboratories for all four materials was extremely poor, as illustrated in figure 7 with results for the nickel-base superalloy Nimonic 101. The results given in figure 7 are in the form of a box plot; thus although only three strain ranges have been used, each is expanded for the purpose of identifying the relatively small repeatability scatter within each laboratory. Figure 7 underestimates the magnitude of the data-scatter because Thomas & Varma had to leave out datasets whenever their analysis showed that the detailed guidelines covering test conditions and reporting requirements had not been followed. Since repeatability scatter was small compared to reproducibility scatter and testpiece samples had been issued randomly to minimize effects due to material-scatter, the problem was clearly one of inadequate testing. By detailed argument, Thomas & Varma (1992) reduced the large number of conceivable reasons for the poor reproducibility to a possible three: (i) bending of the testpiece; (ii) uncertainties in the measurement of strain range; (iii) uncertainties in the measurement and control of temperature.

LCF data are plotted in the engineering literature with lifetime, N_f, as a function of total strain range, $\Delta\epsilon$. Over small strain ranges, the following empirical law adequately approximates behaviour:

$$N_f \Delta\epsilon^{\beta} = K, \tag{3.1}$$

where K and β are constants for a given material only.

Equation (3.1) can be differentiated to give the lifetime uncertainty, dN_f/N_f:

$$\frac{dN_f}{N_f} = -\beta\left[\frac{d\Delta\epsilon}{\Delta\epsilon}\right]. \tag{3.2}$$

Over larger strain ranges, K and β are found to vary systematically, demonstrating that equation (3.1) does not represent the correct physics; a more physically realistic model will be used in § 3 *b*.

Contributions to the uncertainty in strain range $d(\Delta\epsilon)/\Delta\epsilon$ can occur from

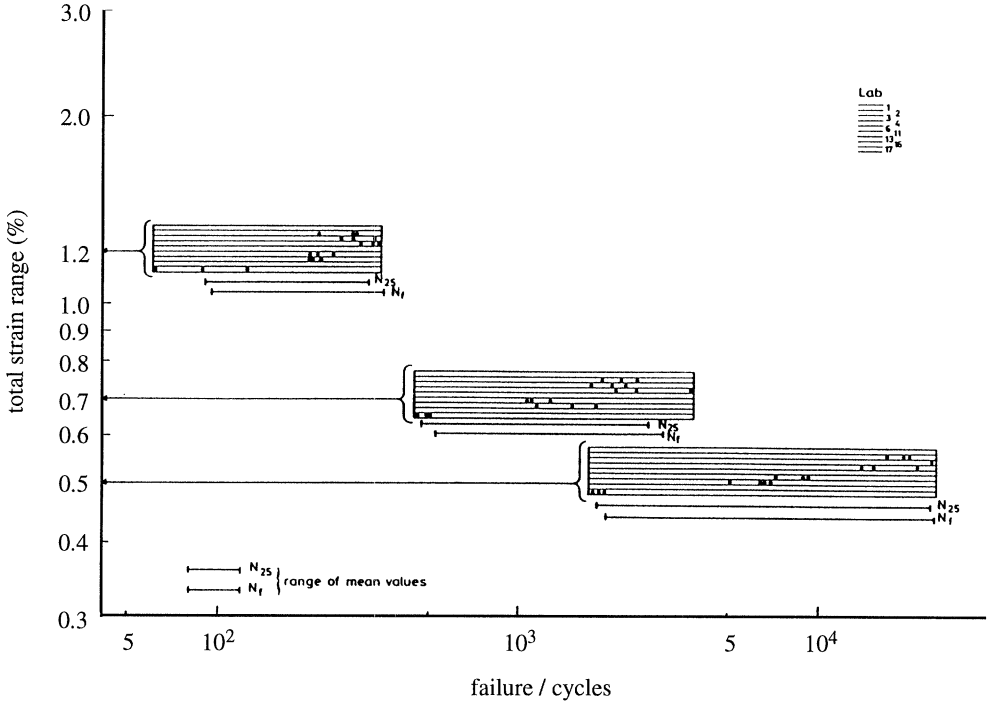

Figure 7. Illustrating the extent of interlaboratory scatter in the BCR round robin exercise, using Nimonic 101 at 850 °C as an example (Thomas & Varma 1992).

measurement-errors alone but these are unlikely to be of any great significance when modern contact extensometers are used correctly. Kandil & Dyson (1993*a*, *b*) considered in considerable detail the influence of testpiece-bending on LCF lifetimes and proposed two new mechanisms that would lead to uncertainties in the value of the control strain range. The major contributing mechanism of testpiece-bending was suggested to be due to a lateral offset of the centrelines of the load train with respect to the machine's frame. This 'load-train-offset' model of bending predicted not only a linear increase in bending strain with load, but a reversal in curvature of the (induced) banana-shaped testpiece between tension and compression loading. As a consequence, the uncertainty in strain range becomes twice the magnitude of the maximum fractional bending strain, $\epsilon_b/\Delta\epsilon_c$, measured in the setting-up procedure. The maximum bending strain ϵ_b is defined as half the difference between the maximum and minimum strains measured across the plane of maximum bending; $\Delta\epsilon_c$ is the control strain range of the test. For a test set up according to ASTM Standard E606-80 (1980), the maximum uncertainty in strain range becomes 10%, rather than 5%, which manifests itself in a potentially larger repeatability-scatter within a single laboratory. The other proposed new source of strain uncertainty is specific to the now almost universal use of a single side-entry extensometer to measure and control displacements and leads to reproducibility errors. Because of the procedures used in LCF testing (bending being invariably measured with a separate strain-gauged testpiece) the location of the extensometer on the testpiece will be randomly related to the plane of maximum strain, induced by the bending. This means that even though the control strain may be set accurately at a constant value in a series of tests, resultant

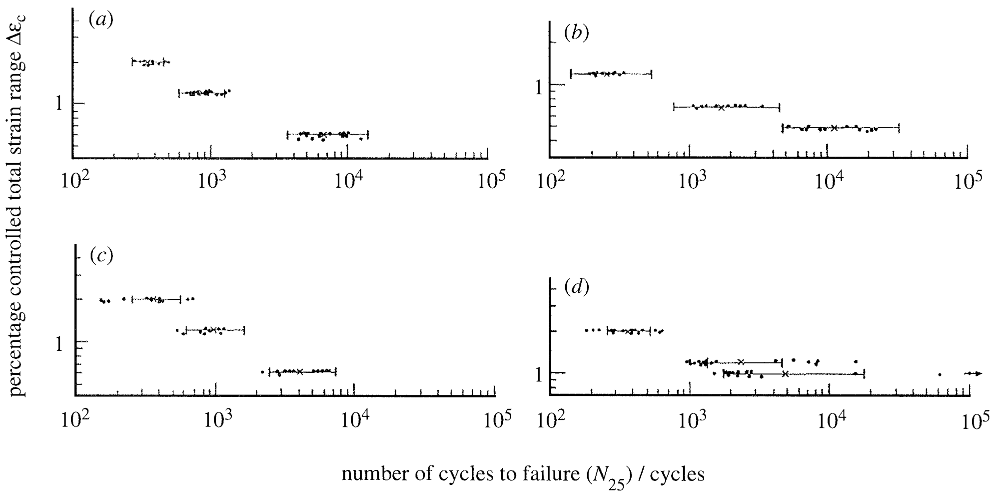

Figure 8. Comparison of predicted scatter in LCF lifetimes with BCR datasets conforming to the ASTM bending criterion (Kandil & Dyson 1993*b*).

lifetimes will depend on the angular position of the extensometer around the circumference of the testpiece, even when bending is kept constant. For example, when the extensometer is positioned in the plane of maximum bending, the maximum strain range, $\Delta\epsilon_{max}$, is equal to the control strain range, $\Delta\epsilon_c$; whereas, when the extensometer is positioned diametrically opposite, $\Delta\epsilon_{max} = \Delta\epsilon_c + 4\Delta\epsilon_b$. Since test data are compared on reported values of $\Delta\epsilon_c$ and yet failure is often thought to initiate at the point of maximum strain, it is clear that the use of a single extensometer will lead to interlaboratory differences in lifetime. To quantify the consequences for interlaboratory scatter, Kandil & Dyson (1993*a*) made the reasonable assumption that the angular position of the extensometer relative to the plane of maximum bending would remain sensibly constant from test to test within a single laboratory (provided that only a single machine were used). The maximum uncertainty in the strain range between laboratories conforming to ASTM Standard E606-80 (1980) now becomes 20% ($4\epsilon_b$). Figure 8 is taken from Kandil & Dyson (1993*b*) to illustrate the reasonably good agreement between their predicted reproducibility-scatter and data obtained from several laboratories participating in the BCR exercise. The data selected for use in figure 8 was a fraction of the whole database, restricted by the constraint that each laboratory should provide evidence that bending conformed to ASTM E606-80. Many data were excluded for this reason, from which it is reasonable to infer that this inability to control the test procedure contributed significantly to the larger scatter of the whole database.

Kandil & Dyson (1993*b*) predicted the scatter-bands in figure 8 by using information derived from the BCR round robin experimental datasets: values of β and K (equation (3.1)) were determined for each strain range from curves of the type shown in figure 6. Lifetime scatter-bands at a prescribed total strain range, such as those presented in figure 8 were calculated using equation (3.1) with the appropriate levels of uncertainty predicted by the 'load-train-offset' model of bending. The procedure demonstrated the potential of the 'load-train-offset' model in understanding scatter in LCF data but its reliance on specific experimen-

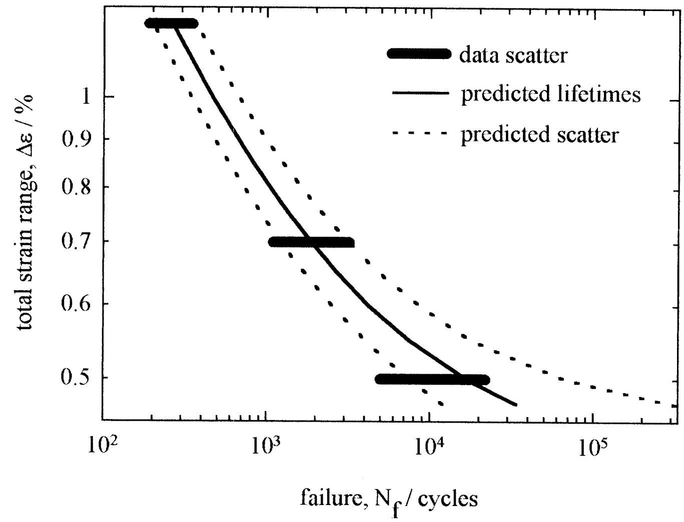

Figure 9. Comparison of LCF data-scatter for Nimonic 101 at 850 °C with lifetime and associated scatter-band predictions calculated from equation (3.5).

tal data precludes general applicability. An alternate and more general procedure, explored in the following section, is to use the 'load-train-offset' model of bending in conjunction with a material-behaviour model and data from independent sources.

(*b*) *A model-based prediction of LCF scatter-bands*

Although complex empirical constitutive models of material behaviour under reversed loading, similar in construction to equation-set (2.1), have been aired in the literature, their detailed application to nickel-base superalloys is still debatable. A very simple material-behaviour model will be used here: uniaxial loading; no transients; no time-dependency; and linear cyclic hardening. The stress range, $\Delta\sigma$, in the LCF cycle is then related to the plastic strain range, $\Delta\epsilon_{\mathrm{p}}$, by:

$$\Delta\sigma = \Delta\sigma_y + h\Delta\epsilon_{\mathrm{p}}, \tag{3.3}$$

where $\Delta\sigma_y$ and h are respectively, the material's cyclic yield stress and work-hardening coefficient.

Rather than using equation (3.1), material failure in LCF is better represented by relating lifetime to plastic strain range (Tavernelli & Coffin 1959):

$$N_{\mathrm{f}}\Delta\epsilon_{\mathrm{p}}^{\nu} = C, \tag{3.4}$$

where C and ν are constants, approximately independent of material. For predictive purposes in engineering, equation (3.4) has to be put in terms of the total strain range $\Delta\epsilon$, controlling the test. Using equations (3.3) and (3.4) and $\Delta\epsilon = \Delta\epsilon_{\mathrm{p}} + \Delta\sigma/E$, where E is Young's modulus, gives

$$N_{\mathrm{f}}\left[\frac{E}{E+h}(\Delta\epsilon - \Delta\epsilon_y)\right]^{\nu} = C, \tag{3.5}$$

where $\Delta\epsilon_y$ is the strain range at yield.

Equation (3.5) has been used to construct figures 9 and 10, which plot the predicted number of cycles to failure, N_{f}, at total strain range, $\Delta\epsilon$ (thin solid

Table 2. *Model parameters used in equation (3.5) to construct lifetime and associated scatter-bands in figures 9 and 10*

v	C	h/MPa	$d(\Delta\epsilon)/\Delta\epsilon$	$\Delta\epsilon_y$
2	10^{-2}	$0.3E$	± 0.1	4×10^{-3} (Nim 101 at 850 °C) 9×10^{-3} (IN 718 at 550 °C)

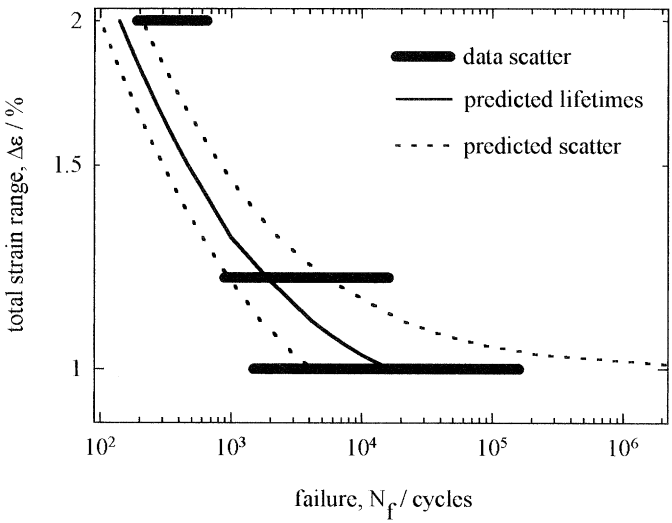

Figure 10. Comparison of LCF data-scatter for IN 718 at 550 °C with lifetime and associated scatter-band predictions calculated from equation (3.5).

line) and the associated scatter-bands (dashed lines) for Nimonic 101 at 850 °C and IN 718 at 550 °C, respectively. The BCR data, plotted as thick horizontal lines are the same as those shown in figure 8. The model parameters are shown in table 2.

The values used for the hardening parameter, h, and the yield strains, $\Delta\epsilon_y$, were deduced from experimental data on the two alloys given by Thomas & Varma (1992): there was much scatter in these stress/strain data and so the values quoted are only approximate. The value of $\nu = 2$ is the same as that reported by Tavernelli & Coffin (1959) for a different set of alloy systems. Strictly, the uncertainty in the strain range, $d(\Delta\epsilon)/\Delta\epsilon$, should be used as a range term (operating only to *reduce* model-based predicted lifetimes) and not as a '±' term. However, since the model parameters have been derived from averaged data and the material behaviour parameters from approximate data, this seems justifiable. A value of ±10% has been used since the maximum uncertainty range predicted by the 'load-train-offset' model in § 3 *a* is 20%, assuming that ASTM Standard E606-80 (1980) is obeyed.

Figures 9 and 10 demonstrate that this simple model has all the necessary components for successfully predicting lifetime and scatter, but requires fine-tuning to get detailed agreement between experiment and theory. However, fine-tuning is hardly worthwhile because the main message concerns the inevitability of large deterministic scatter bands when the control strain range is reduced towards that of the yield strain range. Such deterministic scatter is in addition to probabilistic scatter associated with the intrinsic inability to define yield stress precisely in a polycrystal.

4. Discussion and conclusions

Scatter in mechanical property data has always posed a problem for designers of load-bearing components and has been accounted for historically by so-called factors of safety. A more descriptive term would be factors of ignorance since their magnitudes evolved, quite properly, through experience of disastrous failures. Factors of safety also reflect early inadequacies in the design process and should now be an anachronism in an age when lifetime prediction of complex components using finite element analysis (FEA) is a reality although not yet by any means common-place. The limitation on FEA lifetime prediction lies not in computing power but in (i) improving the physical basis for quantitatively understanding materials' behaviour so that adequate constitutive equations can be developed and (ii) ensuring that the chosen material has been manufactured consistently and the properties measured to an adequate precision. Point (ii) has been addressed in this paper using creep and LCF as case studies, although 'adequate precision' never seems to have been quantified by structural designers.

The paper has attempted to put a scientific framework around this area which, despite its fundamental importance to efficient and cost-effective structural design, has received relatively little attention. Perhaps in the past, steady advances in materials processing and performance have enabled the design engineer to persist in his use of 'factors of ignorance', but an era of 'materials-limited design' can now be foreseen in power engineering applications and this will encourage a better use of the existing materials resource. The continuing dramatic fall in the cost of computing power when offset against the spiralling cost of prototype testing has similarly tilted the balance in favour of better quality databases or, at least, databases of known pedigree. An example of the usefulness of good pedigree data is the creep case-study where the understanding and potential predictability of materials-scatter was enabled only by the systematic experimental work of Toft & Marsden (1961).

The disappointing quality of the interlaboratory data arising out of the BCR round robin exercise on high-temperature LCF has been rationalized by Thomas & Varma (1992) as due to certain inadequacies in the test method. Whether the necessary improvements in testing practice will emerge in the near future remains to be seen, but the work of Kandil & Dyson (1993*a*, *b*) has quantitatively accounted for a large fraction of the scatter and provided guidance on the experimental path to follow to effect such improvements. Perhaps one of the most important conclusions to emerge from the analysis is that large deterministic scatter-bands are inevitable as the cyclic yield strain is approached. This is just the region of most interest to the design engineer and any scatter is usually attributed to materials processing. Perhaps a rethink is required in the whole area of usage in design of data from strain-controlled LCF tests at high temperatures!

Dr S. Osgerby (NPL) kindly supplied digitised data of creep lifetime as a function of stress, derived from the paper by Toft & Marsden (1961). Comments by Dr F. A. Kandil (NPL) on a draft of the manuscript have been incorporated into the final text.

PHILOSOPHICAL TRANSACTIONS OF THE ROYAL SOCIETY A MATHEMATICAL, PHYSICAL & ENGINEERING SCIENCES

References

Ashby, M. F. 1989 Materials selection in conceptual design. In *Materials and engineering design* (ed. B. F. Dyson & D. R. Hayhurst), Part I, ch. 2, London: Institute of Metals

Ashby, M. F. 1991 Materials and shape. *Acta metall. Mater.* **39**, 1025–1039.

ASTM Standard E606-80 1980 Standard recommended practice for constant-amplitude low-cycle fatigue testing. *1991 Annual Book of ASTM Standards*, Part 3.01, pp. 609–621, Philadelphia, PA: American Society for Testing and Materials.

Dyson, B. F. & Osgerby, S. 1993 Modelling and analysis of creep deformation and fracture in a 1Cr1/2Mo ferritic steel. *NPL Rep.* DMM(A)116

Dyson, B. F., Loveday, M. S. & Gee, M. G. 1995 Materials metrology and standards: an introduction. In *Materials metrology and standards for structural performance* (ed. B. F. Dyson, M. S. Loveday & M. G. Gee), ch. 1. London: Chapman and Hall.

Dyson, B. F. & McLean, M. 1990 Creep deformation of engineering alloys: developments from physical modelling. *ISIJ Int.* **30**, 802–811.

Gould, D. & Loveday, M. S. 1992 A reference material for creep testing. In *Harmonisation of testing practice for high temperature materials* (ed. M. S. Loveday & T. B. Gibbons), ch. 6. London: Elsevier Applied Science.

Henderson, W. T. K. & Thomas, G. B. 1995 Accredited testing and reference materials. In *Materials metrology and standards for structural performance* (ed. B. F. Dyson, M. S. Loveday & M. G. Gee), ch. 10. London: Chapman and Hall.

Hossain, M. K. & Sced, I. R. 1995 Metrology for engineering materials. In *Materials metrology and standards for structural performance* (ed. B. F. Dyson, M. S. Loveday & M. G. Gee), ch. 9. London: Chapman and Hall.

Kandil, F. A. & Dyson, B. F. 1993*a* The influence of load misalignment during uniaxial low cycle fatigue testing. I. Modelling. *Fatigue Fract. Engng Mater. Struct.* **16**, 509–527.

Kandil, F. A. & Dyson, B. F. 1993*b* The influence of load misalignment during uniaxial low cycle fatigue testing. II. Applications. *Fatigue Fract. Engng Mater. Struct.* **16**, 529–537.

Lifshitz, J. M. & Slyozov, V. V. 1961 *J. Phys. Chem. Solids* **19**, 35–50.

Lin, J., Hayhurst, D. R. & Dyson, B. F. 1993*a* The standard uniaxial testpiece: computed accuracy of creep strain. *J. Strain Analysis* **28**(1), 20–34.

Lin, J., Hayhurst, D. R. & Dyson, B. F. 1993*b* A new design of uniaxial creep testpiece with slit extensometer ridges for improved accuracy of strain measurement. *Int. J. Mech. Sci.* **35**(1), 63–78.

Loveday, M. S. 1992 Towards a tensile reference material. In *Harmonisation of testing practice for high temperature materials* (ed. M. S. Loveday & T. B. Gibbons), ch. 7. London: Elsevier Applied Science.

Manson, S. S. & Haferd, A. M. 1953 A linear time–temperature relation for extrapolation of creep and stress rupture data. *Nat. Advisory Committee Aeronautics Tech. Note* 2890.

McLean, M., Dyson, B. F & Ghosh, R. N. 1991 Recent evolution of gas turbine materials and the development of models for life prediction. In *Mechanical behaviour of materials. VI* (ed. M. Jono & T. Inoue), vol. 12, pp. 49–58. Pergamon Press.

Tavernelli, J. F. & Coffin, L. F. 1959 A compilation and interpretation of cyclic strain fatigue tests on metals. *Trans ASM* **51**, 438–453.

Thomas, G. B., Hales, R., Ramsdale, J., Suhr, R. W. & Sumner, G. A. 1989 A code of practice for constant-amplitude low cycle fatigue testing at elevated temperature. *Fatigue Fract. Engng Mater. Struct.* **12**, 135–153.

Thomas, G. B. & Varma, R. K. 1992 Review of the BCR/VAMAS low cycle fatigue intercomparison programme. In *Harmonisation of testing practice for high temperature materials* (ed. M. S. Loveday & T. B. Gibbons), ch. 8. London: Elsevier Applied Science.

Toft, L. H. & Marsden, R. A. 1961 The structure and properties of 1Cr1/2Mo steel after service in CEGB power stations. In *Structural processes in creep*, 276–294. London: Institute of Metals.

Discussion

P. Hirsch (*Department of Materials, University of Oxford, UK*). I am deeply concerned about the quality of the data quoted on low cycle fatigue. The spread of the results from different laboratories exceeds the safety factor of 25 usually applied in safety assessments to allow for various uncertainties. This is potentially serious because when making such assessments data from different laboratories, which might indicate the presence of systematic errors, are not always available. It is to be hoped that the efforts of those engaged in BCR and other projects will soon lead to a tightening of the specifications governing measurement techniques, so that these systematic errors can be reduced to negligible values.

B. F. Dyson. I hope that Sir Peter Hirsch's concern will soon be allayed. My former colleague at NPL, Dr F. A. Kandil, is now leading an investigation sponsored by BCR and aims to develop a theoretical and experimental framework for quantifying uncertainties in low cycle fatigue lifetime data due to errors in measurement during testing. The ultimate objective is to provide a robust methodology that will reduce interlaboratory data-spread to a factor less than about five in lifetime. Although this is not negligible as Sir Peter clearly wishes, it is still an extremely ambitious target because of the difficulties inherent in this test.

M. McLean (*Imperial College, London, UK*). Professor Dyson's model incorporates the effect of the coarsening of a fixed volume fraction of strengthening particles, and it clearly gives a good representation of the high-temperature behaviour of this class of alloy. In others there can be significant changes in phase chemistry. For example, we have recently shown in a 12CrMoNbV steel that MC transforms to $M_{23}C_6$ during service. This reduction in the volume fraction of MC leads to a reduction in strength.

R. C. Thomson (*Cambridge University, UK*). Professor Dyson described the data presented (Toft & Marsden) by a single coarsening parameter, S. However, the particles present in the $1Cr\frac{1}{2}Mo$ steel microstructure undergo transformations from cementite to alloy carbides M_7C_3 and $M_{23}C_6$, etc. It is known in the literature that these Cr rich alloy carbides coarsen much more slowly than cementite. Could he comment on how these transformations and subsequent changes in coarsening rate might be incorporated into the coarsening parameter, and secondly on whether he thinks there is a need to do so in terms of explaining the macroscopic creep behaviour?

B. F. Dyson. Professor McLean and Dr Thomson state quite correctly that the present model is concerned only with the effect on creep rate when a spherical particulate dispersion of fixed volume fraction coarsens according to the Lifshitz–Slyozov equation. Real materials are usually more complicated and this is particularly the case with low-alloy ferritic steels, which is the point of commonality between the two comments. A succession of coarsening carbides could in principle be incorporated into the present model by introducing a damage parameter with its appropriate evolution equation for each carbide. However, it is debatable whether the effort involved in producing such a sophisticated model would be worthwhile, except as an academic exercise. A better first attempt in my view is to assume the dominance of a single carbide particle for all but the shortest times, particularly since one important aim of developing physically based constitutive equations is to predict behaviour at stresses giving lifetimes of the order 10^5 h.

PHILOSOPHICAL TRANSACTIONS OF THE ROYAL SOCIETY A MATHEMATICAL, PHYSICAL & ENGINEERING SCIENCES

Professor McLean suggests that in the currently important 12CrMoNbV steel, the reduction in volume fraction with time of one of the carbides is life-limiting. In such a situation, the Lifshitz–Slyozov coarsening relationship in equation-set (2.1) would need to be replaced by one containing the evolution rate of volume fraction, with suitable changes also being made to the strain-rate equation to accept volume fraction as a damage parameter.

M. S. Loveday (*Division of Materials Metrology, National Physics Laboratory, Teddington, UK*). Professor Dyson has highlighted in a very clear manner the importance of assessing measurement uncertainty, and, in particular, the role of modelling a material's behaviour to provide a clearer means of identifying the causes of scatter in experimental data.

The need for a statement of uncertainty of measurement is now a requirement for International (ISO) and European (CEN) testing standards; in addition accreditation agencies such as NAMAS (National Measurement Accreditation Service) now require an assessment of uncertainty of measurement, whether it be for determining material properties, or for any other physical, chemical or electrical measurments. A recently published document – 'A guide to the expression of uncertainty in measurement' – has been issued by ISO, IEC, OIML and BIPM which provides an approach for assessing total uncertainty. Although this document is comprehensive and based on sound statistical methods, it is difficult to see how the majority of technical staff in testing laboratories would be able to understand and implement its recommendations; however, simplified guidelines in the UK are being prepared by NAMAS and the BMTA (British Measurement and Testing Association).

B. F. Dyson. I agree with the sentiments expressed about the document; its incomprehensibility certainly extends to me. I wish NAMAS and BTMTA well in their endeavour to provide us all with a translation!

PHILOSOPHICAL TRANSACTIONS OF THE ROYAL SOCIETY A MATHEMATICAL, PHYSICAL & ENGINEERING SCIENCES