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SOME NOVEL TEST METHODS FOR, AND ASSOCIATED PROBLEMS OF, MECHANICAL STRENGTH CHARACTERISATION OF ENGINEERING CERAMICS

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ABSTRACT

The development of ceramic components for structural purposes in internal combustion engines, or gas turbines, will rely on accurate and reliable design data. A prime requirement will be information on mechanical strength under a variety of loading conditions and environments.

The simplest method of strength assessment is the Modulus of Rupture (MOR) via a three or four point bend test. However, the effective volume of material examined in each test is very small, and possibly, this type of test may be best used for the study of surface and near-surface behaviour.

Therefore, there is a need for tests which are capable of examining large volumes of material, preferably economical in terms of equipment cost and test time, since there is still a need to characterise material on a statistical basis.

This paper describes methods of:-
(a) Multiple bend tests in one "furnace load" at elevated temperature,
(b) A relatively inexpensive elevated temperature axial tensile test system and
(c) The development of an axial test system capable of complex thermo-mechanical cyclic loads.

INTRODUCTION

The commercial pressures for more competitive air transport produces a continual exploration for effective designs and new materials: the latter will, of course, have some considerable effect on design and, eventually, the vehicle performance.

Focusing on the gas turbine propulsion units: the demand is for improved power-to-weight ratio and specific fuel consumption. These improvements can be obtained through improved combustion, higher operational temperatures and the elimination of expensive cooling air.

Without cooling air, the superalloys cannot operate in the temperature range of 1300°C to 1600°C and for this reason Rolls-Royce is developing the use of silicon nitride and silicon carbide ceramics for high temperature turbine components (Fig.1).

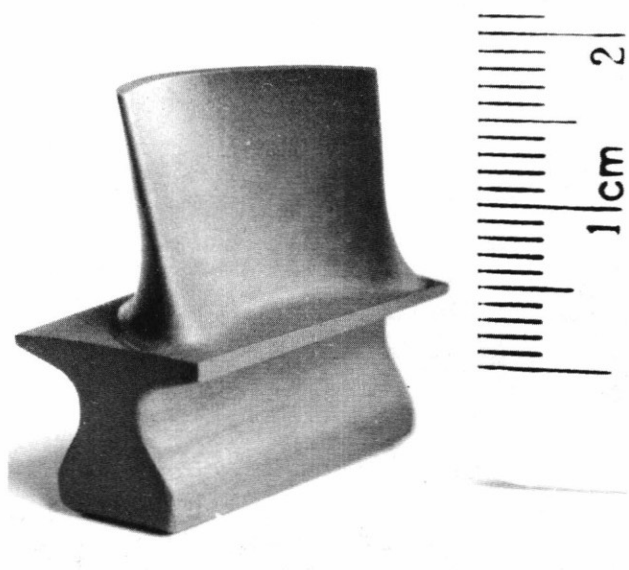

FIGURE 1 Silicon Nitride H.P. Turbine Blade

These materials have some attractive high temperature properties:-
- High strength
- Creep resistance
- Oxidation resistance
- Thermal shock resistance

Last, but not least, a further attraction for these materials is a significantly lower density than the superalloys.

However, a disadvantage with the monolithic form of these materials is that they are inductile with a K_{IC} of 5 - 10 MPa$\sqrt{m}$. This inductility is reflected in defect sensitivity, which is further complicated by the defect distribution mainly introduced during fabrication and finish machining.

This means that the strength characterisation has to be carried out on a statistical basis, and for the results acquired in this paper Weibull statistics have been used together with a modified Weibull equation to account for "size-effect".[1]

In regard to the test methods to be discussed:- first, the bend test: historically, three and four point bend tests have always been favoured for material screening purposes, mainly because they are relatively inexpensive and easy to manufacture.

In order to produce a reasonable number of test results with economy in equipment and time, the Leavesden Laboratories of Rolls-Royce have produced a simple means of short term sequential tests of five specimens in one "furnace load".

Next, a further development at the Leavesden Laboratories is a relatively inexpensive tensile test system for elevated temperature, which is also being developed for tension-tension fatigue and thermo-mechanical test cycles. The trend, as it can be seen, is to develop more realistic tests for material assessment.

TEST REQUIREMENTS FOR CERAMIC MATERIALS

Generally speaking material behaviour is examined under "zero-time" or "real-time" conditions. The "zero-time" tests are usually carried out as a means of screening various material formulations, surface finishes or coatings.

The "real-time" tests are used to examine stress rupture, creep, fatigue and combinations of thermal and mechanical fatigue. All these supply information for the basis of life prediction and behavioural modelling.

Zero Time Testing

Bend Tests This mode of strength test, up to the present time, is probably the most popular: the test specimens and rigs are relatively easy to manufacture. At ambient temperature, there is generally no problem in carrying out numbers of tests which meet the statistical requirements for material assessment within a few hours. However, this is not the case at elevated temperature, especially when resources are limited. With this in mind, the Leavesden Laboratory devised what might be described as a ceramic ladder, wherein the rungs of the ladder are the test specimens which are subjected to sequential fracture.

The system in more detail is as follows:- A series of five reaction bonded silicon nitride (RBSN) hollow cylinders are mounted on top of one another on a platform on the actuator of an axial servo-screw driven tension/compression testing machine: concentricity is maintained by mating peripheral recesses in the cylinders. The test specimens are arranged diametrically across the cylinders; they are mounted on rollers which are located in "V" shaped recesses cut across the chords of the cylinders (Fig.2). The third point is a silicon carbide plunger mounted on the load cell end which carries the controlling and monitoring thermocouples to be positioned in close proximity to each specimen. Each assembly is pushed up in turn to the plunger to apply the load. A split furnace encases the assembly with the hot zone produced by Crusilite heating elements; the arrangement can be seen in Fig.3.

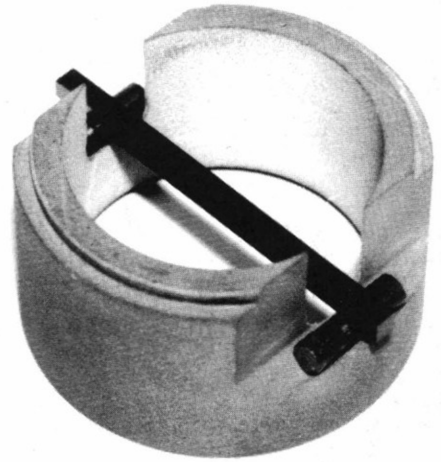

FIGURE 2 Loading Ring used in the Elevated Temperature, Three Point Bend Rig

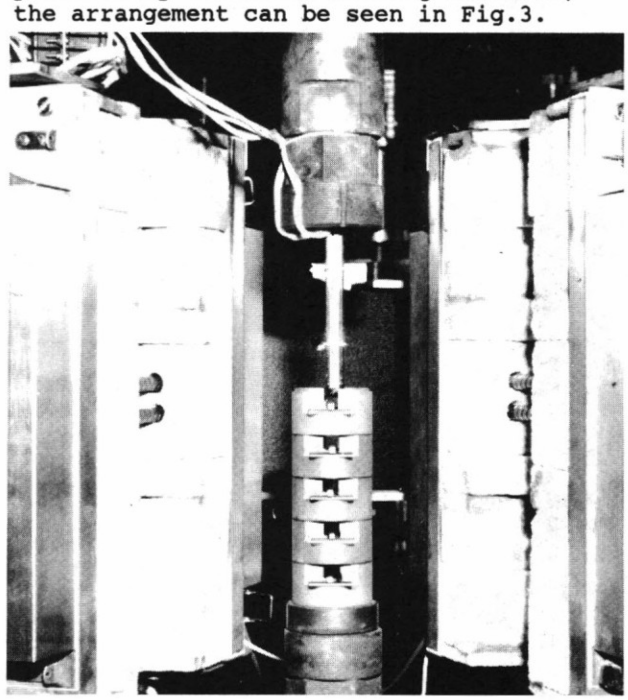

FIGURE 3 Assembly of the Elevated Temperature Bend Rig

This system has been used to measure the MOR strengths of many hundreds of specimens up to 1400°C in air. Generally three furnace load can be tested per working day when operating up to 1200°C but, due to limitations in furnace design, the machine utilisation is somewhat less at higher temperatures.

Axial Tensile Testing Although it has been shown by Stanley et al, Hattori et al and Matsusu et al[1,2,3] that bend and tensile tests of differing specimen sizes can be related by the Weibull Volume Concept (see below). The frictional restraints occurring at the loading points at elevated temperatures on three or four point bend tests will produce errors in the measured strength. Also, because of the stress gradients produced, there will always be arguments about the significance of the volume of material examined during bend tests.

Therefore it was decided to develop axial tensile testing. At first, ceramic materials were considered for the construction of the load train. But this was dismissed on the basis of manufacturing complications, cost and relatively short life expectancy. A design philosophy involving conventional nickel-based superalloys, with water cooling and aided by ceramic heat shields, was adopted (Fig.4).

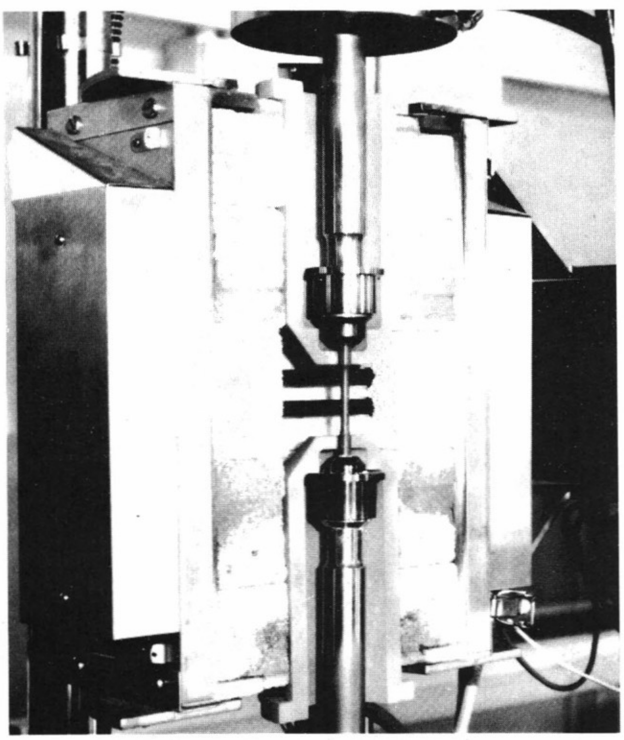

FIGURE 4 Assembly of the Elevated Temperature Axial Tensile Rig

Button-ended cylindrical specimens are used to facilitate an accurate location within precision ground split collets. These are coupled via hemispherical seatings to the ends of water cooled shackle columns, axiality is further enhanced by the attachment of low friction universal joints between the shackle train and the testing machine. Contact stresses between the split collets and the specimen button head are alleviated by a boron nitride powder cushion. To reduce the bending component produced by misaligned "sticking" at coupling points, a cyclic, low amplitude "shake-down" load is applied, subsequent results obtained from strain gauged specimens indicates that this procedure reduces the bending component to less than 1%.

It was considered that the best way to test the axiality of the system; and the specimen design, was to test the material under the most inductile conditions. Therefore, the proving trials for these shackles were carried out at room temperature using a silicon nitride material containing a crystalline grain boundary matrix, which had been previously well characterised in bending over a wide range of temperatures.

The tests were carried out on three different test volumes:- 210, 375 and 750 mm^3. The results from these different axial test volumes were to be compared with each other, and also with bend test results from the same material. The surface finish was similar on all specimens: namely about 0.1μm C.L.A.. The direction of grinding of the axial specimens was transverse with respect to the applied stress, and on the bend specimens, both longitudinal and transverse directions. For comparison purposes, because of the different modes of test and specimen sizes, the Unit Strength Concept[1] and Weibull Volume Concept were employed, i.e.:-

$$\frac{\sigma_{v1}}{\sigma_{v2}} = \left[\frac{V_2}{V_1}\right]^{1/m} \qquad (1)$$

where V_1 and V_2 are the effective volumes (derived from the stress volume integrals) of a test specimen or a component, and, σ_{v1} and σ_{v2} are the stresses associated with the same probability of survival. In these trials all failures occurred at random in the test section with 90% of the failures initiating at the surface. The rupture strengths are shown in Table 1.

TABLE 1

Volume (mm^3)	No. Res.	Mean σ (MPa)	σ/Unit Vol (MPa/cm^3)	Weibull Modulus
750	8	217.4	209.3	7.66
375	10	237.9	200.6	5.70
210	10	251.2	217.0	10.56

It can be seen that the unit strength is reasonably similar for all three test volumes. However, the correlation between the bend and axial tests is not clear as can be seen in Table 2.

TABLE 2

Specimen Cond.	**No. Res.**	**Mean σ (MPa)**	**σ/Unit Vol (MPa/cm^3)**	**Weibull Modulus**
Long. Ground	47	825.9	315.4	7.70
Trans. Ground	50	458.8	151.4	6.89

It is the unit strength for the transverse ground conditions which are vaguely similar, and as a reminder: the cylindrical specimens are transverse ground, but without the "edge" condition that would be found with the rectangular specimens.

The disturbing fact about these results was:-

(a) The actual grinding direction for conformal shapes on structural components is transverse e.g. Fig 5.

(b) Because of the crystalline nature of the grain boundary matrices, the grinding damage could not be annealed by conventional thermal processes.

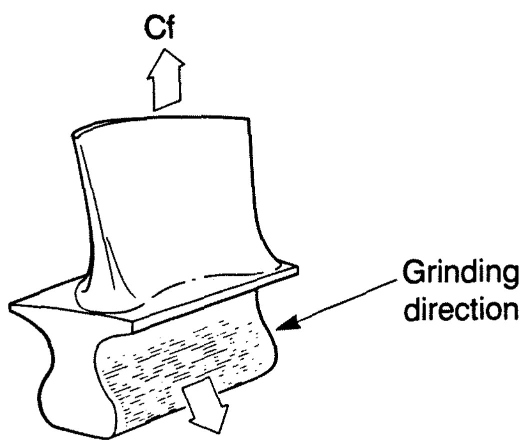

FIGURE 5 Schematic of the Grinding Direction on a Turbine Blade Root

Ion implantation provided the source of palliation: after some experimentation the critical dose was about 10^{17} nitrogen ions per cm^3 at 90keV. It can be seen from Table 3 that the base line properties were completely restored.

TABLE 3

Specimen Cond.	**No. Res.**	**Mean σ (MPa)**	**σ/Unit Vol (MPa/cm^3)**	**Weibull Modulus**
Trans. Ground+ Ion Imp.	25	867.4	447.1	13.31

Recent work has shown that the repair of machining damage coarser than 0.1μm C.L.A. on this type of material requires higher potentials and different ion species (this work will be reported at a later date by R. Quinn and G. Syers).

Axial Tensile Testing at 1400°C

Tests have been carried out at 1400°C using a furnace with Crusilite rods as heating elements. The temperature gradients over the gauge length have been within the British Standards, however, these have been further reduced by modifications to the heat shields, which, to a certain extent, act as heat sinks. The maximum temperature registered on the shackles was 800°C on the outer surfaces the split collets.

During some early high temperature tests it was found that some specimens were failing in the radius adjacent to the button-head, this was found to be due to difficulties in controlling the grinding parameters in this critical region. Once again, ion implantation provided the solution to this problem.

It is interesting to note that these shackles, of simple design: designed in the Leavesden Laboratory and manufactured by an outside contractor: cost £8500; perhaps a quarter of the cost of those available commercially.

Real Time Testing

As it has been stated zero time tests are extremely useful for material characterisation, and if the material, for example: were to be used for the construction of, say, a turbine disc: then this information could be used only to calculate the disc burst criteria.

However, these conditions do not represent the real operating conditions, neither do the so-called "real-time" tests carried out under isothermal conditions. In reality, turbine blades and discs experience both thermal and mechanical fatigue, and the two mechanisms are not necessarily in phase with each other. Because of the nature of these materials at extreme temperatures, it is important, and perhaps ideal, to achieve the behaviouristic understanding within the Laboratory, rather than extended and costly engine trials.

The method chosen to simulate transient and steady-state temperature conditions was Radio Frequency Induction Heating using specially designed coils and susceptors as the heating source.

The heating system and the load controls on the Rolls-Royce system are linked by a microprocessor as a means of controlling the thermal and mechanical conditions (Fig.6).

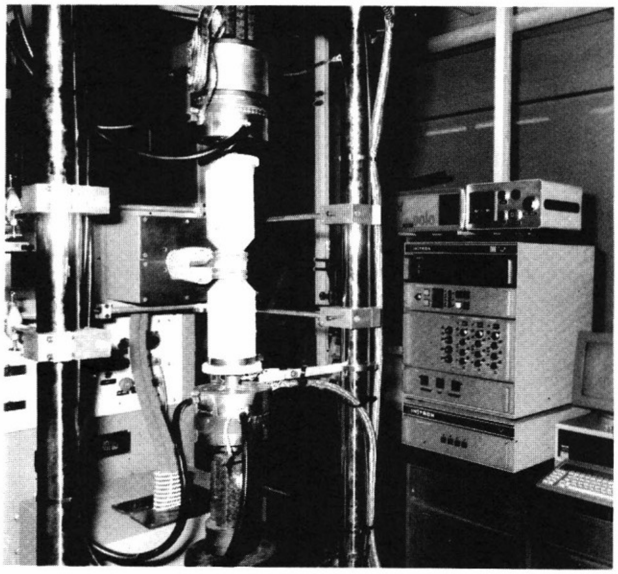

FIGURE 6 The Complex Thermo-Mechanical Cycling Rig

This system is being developed in the Leavesden Laboratories for tension-tension fatigue with in phase and phase-lag heating. In the future it is planned to develop a tension-compression technique together with simplified and less expensive test specimens.

CONCLUSIONS

(a) Bend tests are suitable for surface characterisation an for material screening.

(b) There is a need to radically reduce the friction at the loading points on bend rigs at high temperatures.

(c) Machining damage must always be taken into account or palliated.

(d) Axial tests are probably the most reliable method of material assessment, and not necessarily expensive.

(e) The development of more realistic thermo-mechanical tests within the laboratory could save money on engine trials.

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