JOURNAL OF MATERIALS SCIENCE 14 (1979) 2575–2580

Statistical analysis of the brittle fracture of sintered tungsten

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A Weibull analysis was applied to the fracture data of sintered tungsten round bar specimens. Fracture data were obtained by performing flexural and tensile tests on these components. Two quantities were obtained which characterized the material variability and strength for each test method. The correlation between these quantities for the two test methods was found to be close.

Nomenclature

C	least square line intercept
P_f	failure probability
P_f^i	experimental failure probability
V	specimen volume
W	applied load
X	horizontal coordinate of least square line
Y	vertical coordinate of least square line
d	specimen diameter
L	distance between supporting knife edges
m	Weibull modulus
v	unit volume
σ	stress
σ_0	normalizing stress
σ_{fV}	failure stress of specimen
σ_{fv}	unit volume failure stress of specimen
σ_{fv}^B	unit volume failure stress in bending
σ^i	experimental stress
Γ	gamma function

1. Introduction

Because of its refractory properties, tungsten is used as a throat insert for uncooled nozzles of solid propellant rocket motors whenever the highest possible resistance to erosion is required.

Unfortunately sintered tungsten, unless it is heavily cold worked, as in tungsten wire, generally behaves like a brittle material at room temperature. Although sintered tungsten has a much higher resistance to thermal shock than conventional ceramic materials, under the very high temperature gradients experienced in a rocket nozzle, tungsten throat inserts are frequently observed to contain thermal shock cracks after firing. In some designs the initial cracking due to thermal stress may lead to break-up of the throat insert during firing if adequate support is not provided. An analysis allowing for the statistical variation of crack severity and orientation is therefore needed for the design of safe nozzle inserts.

The bulk and surface of sintered tungsten contain flaws which are distributed randomly as regards both orientation and size. For a given stress there is a variable probability that one of these flaws may become critical and propagate catastropically. The origin of failure is not necessarily the point of highest stress, but depends on the combination of stress level, component volume, flaw size and orientation. Because of the inherent variability of the material nominally identical specimens fail at different loads.

To characterize this type of material a statistical analysis, based on the Weibull distribution [1], is presented which allows for material variability and strength. The Weibull modulus m gives an inverse measure of material variability while another quantity $\bar{\sigma}_{fv}$ denotes the average tensile strength of a unit volume cube of material.

In Section 3 two test methods are described which allow m and $\bar{\sigma}_{fv}$ to be found experimentally. The first is the standard tensile test and the second is the three-point bend test. In the bend test the specimen is supported on knife edges and point loaded centrally. The tensile test is more

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difficult and the specimen tends to fail in bending if it is not perfectly aligned. Stress concentrations may develop at the grips and may lead to premature failure. The bend test has none of these faults.

The fracture data from both tests were analysed according to the theory outlined in Section 2. The failure characteristics m and $\bar{\sigma}_{fv}$ were evaluated for both cases and the experimental agreement was close.

2. Theoretical analysis

2.1. Weibull analysis

For the uniaxial tensile test and the three-point bend test the failure probability P_f at an applied stress level σ may be derived from the simple Weibull distribution [1]

$$P_f = 1 - \exp\{-[\sigma/\sigma_0]^m\}. \quad (1)$$

The Weibull modulus m is an inverse measure of the variability of the material. The term σ_0 has dimensions of stress and is related to the average failure tensile stress $\bar{\sigma}_{fV}$ of a batch of nominally identical specimens by the equation [2]

$$\sigma_0 = \bar{\sigma}_{fV} \Big/ \left(\frac{1}{m}!\right)^{1/m}. \quad (2)$$

The expression $(1/m)!$ is the factorial function of $1/m$ [3].

For the uniaxial tensile test Equation 1 may be written in the form [2]

$$P_f = 1 - \exp\left[-\left(\frac{1}{m}!\right)^m \frac{V}{v}\left(\frac{\sigma}{\bar{\sigma}_{fv}}\right)^m\right]. \quad (3)$$

In Equation 3 use is made of the volume size effect ratio

$$\frac{\bar{\sigma}_{fV}}{\bar{\sigma}_{fv}} = \left(\frac{v}{V}\right)^{1/m} \quad (4)$$

to relate strengths to the unit volume v.

For the bend test the applied stress is uniaxial and non-uniform. Under these conditions Equation 3 generalizes to

$$P_f = 1 - \exp\left[-\left(\frac{1}{m}!\right)^m \frac{V}{v}\left(\frac{\sigma_{max}}{\bar{\sigma}_{fv}}\right)^m S(V)\right]. \quad (5)$$

The quantity σ_{max} is the maximum bending stress defined by

$$\sigma_{max} = \frac{8WL}{\pi d^3} \quad (6)$$

where W denotes the applied load, L the distance between the supporting knife edges, and d the specimen diameter. The term $S(V)$ is a stress volume integral which, for circular specimens under three-point loading, is given by

$$S(V) = \frac{2}{2\pi^{1/2}} \frac{\Gamma\left(\frac{m+1}{2}\right)}{(m+1)\,\Gamma\left(\frac{m+4}{2}\right)}. \quad (7)$$

The gamma function $\Gamma(X)$ appearing in Equation 7 is tabulated for all real values of X and may be readily evaluated [4].

In many situations the strength capability of a brittle material is quoted in terms of its maximum bending stress. If a Weibull analysis based on this quanitity is carried out a strength factor for bending may be defined as $\bar{\sigma}_{fv}^B$. Comparison of Equations 3 and 5 leads to the relationship

$$\frac{\bar{\sigma}_{fv}}{\bar{\sigma}_{fv}^B} = \frac{2}{2\pi^{1/2}} \frac{\Gamma\left(\frac{m+1}{2}\right)}{(m+1)\,\Gamma\left(\frac{m+4}{2}\right)}. \quad (8)$$

2.2. Evaluation of the failure characteristics

The failure probability of a circular rod under tension or bending is given respectively by Equation 3 or Equation 5. Each contains the failure Weibull parameters m and $\bar{\sigma}_{fv}$ and their evaluation for the fracture data is now described.

The specimen fracture loads are ranked in ascending order W_i, $i = 1, 2 \ldots N$. From these loads the failing stresses in tension or bending are evaluated from the respective formulae

$$\left.\begin{aligned} \sigma^i &= \frac{4W_i}{\pi d^2} \\ \sigma^i &= \frac{8W_iL}{\pi d^3} \end{aligned}\right\} i = 1, 2, \ldots N. \quad (9)$$

Associated with these fracture stresses are the experimental failure probabilities [5]

$$P_f^i(\sigma_i) = (i - \tfrac{1}{2})/N, \qquad i = 1, 2, \ldots N. \quad (10)$$

Since the Weibull distribution is assumed to fit the ranked data the plot of

$$\left.\begin{aligned} Y_i &= \log_e \log_e (1/(1-P_f^i)) \\ X_i &= \log_e (\sigma^i) \end{aligned}\right\} i = 1, 2, \ldots N. \quad (11)$$

should lie on the straight line

$$Y = mX + C. \quad (12)$$

If the distribution assumption is correct the slope of this line gives the Weibull modulus m. Unit volume strengths then follow from the respective formlae

$$\bar{\sigma}_{fv} = \left(\frac{V}{v}\right)^{1/m} \left(\frac{1}{m}!\right) e^{-C/m}, \quad (13)$$

$$\bar{\sigma}_{fv} = \left[\frac{V}{v} S(V)\right]^{1/m} \left(\frac{1}{m}!\right) e^{-C/m} \quad (14)$$

for uniaxial tension or bending.

3. Experimental results

Twelve tensile test specimens were machined from a sintered and forged tungsten rocket nozzle, 50 mm diameter and 50 mm long, with the tensile axis parallel to the nozzle axis. The specimens were centreless ground to the dimensions shown in Fig. 1. The tungsten was manufactured by Metallwerk Plansee, Austria to MOD, QAD (Weapons) Specification GW(AIR) 423 and had a density exceeding 95 per cent of the theoretical value and a minimum purity of 99.74 per cent.

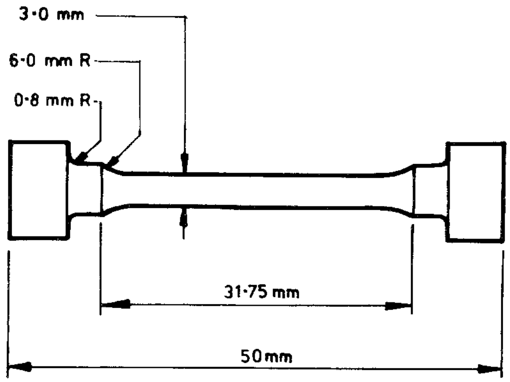


Figure 1 Cylindrical tensile test piece.

The tensile tests were carried out on a standard Instron 10 000 kg testing machine. A cross-head speed of 0.5 mm min^{-1} was selected which, over a gauge length of 32 mm, corresponded to a strain rate of 2.5×10^{-4} sec^{-1}. The tensile properties were derived from the recorded fracture loads indicated on the Instron chart.

The tensile test specimens fractured in a brittle manner. Using a magnification ratio of 100 between chart and cross-head movement, no deviation from elastic behaviour could be detected prior to the onset of rapid fracture. The fracture loads exhibited wide scatter and only half of the specimens broke within the gauge length, as shown in Fig. 2. The others failed prematurely at stresses below yield in the region of the collets in which they were held. Precautions were taken to ensure accurate alignment of the specimen axis with the load axis by using a universal coupling to both

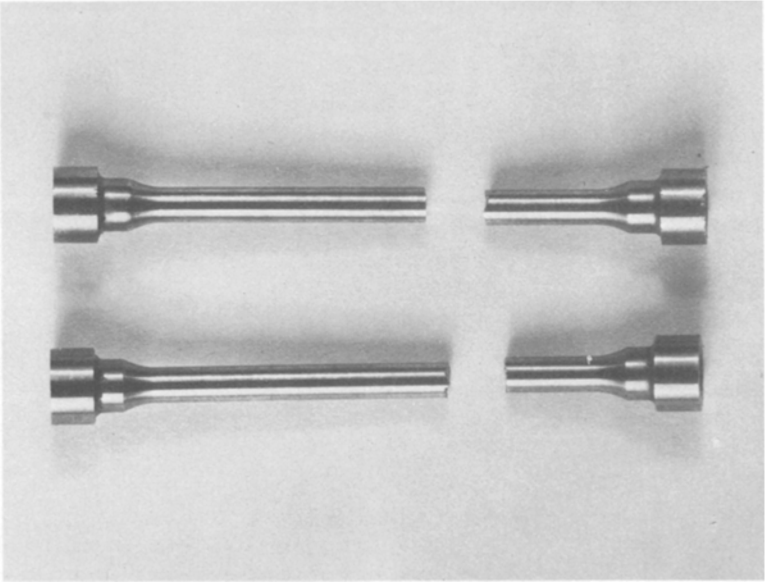


Figure 2 Tensile test specimens showing brittle fracture within the gauge length.

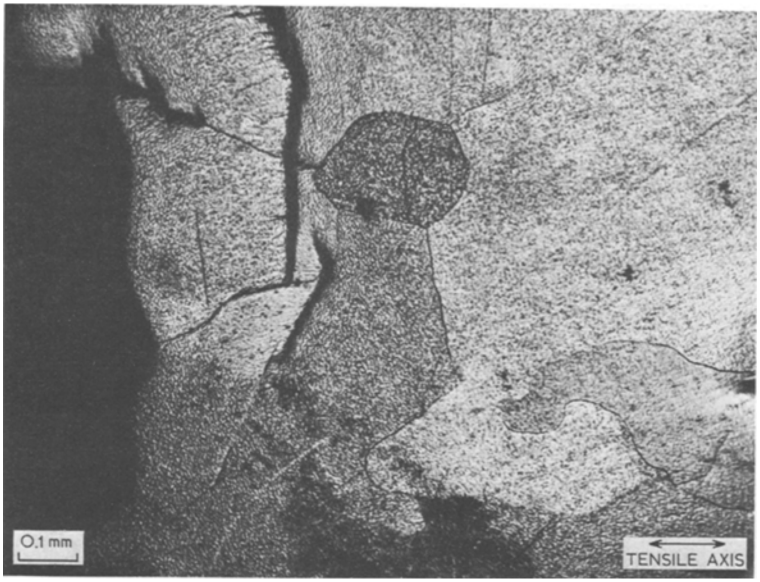
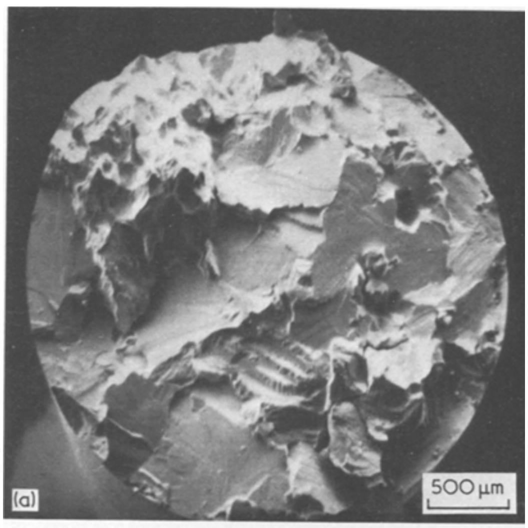
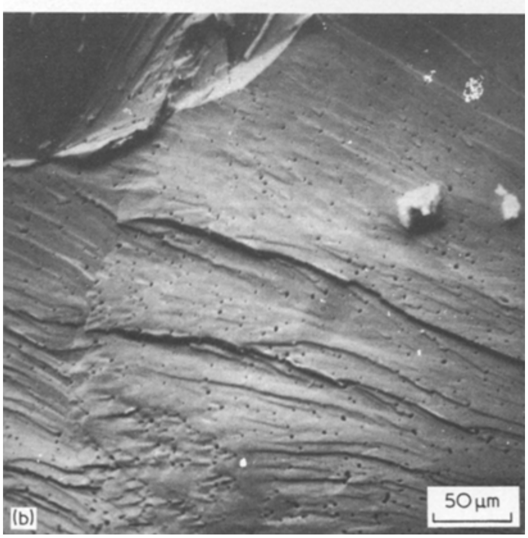


Figure 3 Longitudinal section through fracture surface of tensile specimen showing grain structure and cleavage cracks (etchant: Murakami's reagent).

specimen grips. This allowed each end of the specimen to move laterally with two degrees of freedom while being pulled in the vertical mode. Alignment was improved by slightly pre-loading the specimen and applying a vibrating marker (Vibratool) to both grips until the load dropped to a constant value. This procedure wa epeated until no further drop in load occun.d. It was therefore felt that premature failure was probably caused by stress concentrations becoming critical in the region of the collets and not by poor alignment.

Typical fracture surfaces are shown in Figs. 3 and 4. It can be seen that the grain size is coarse,





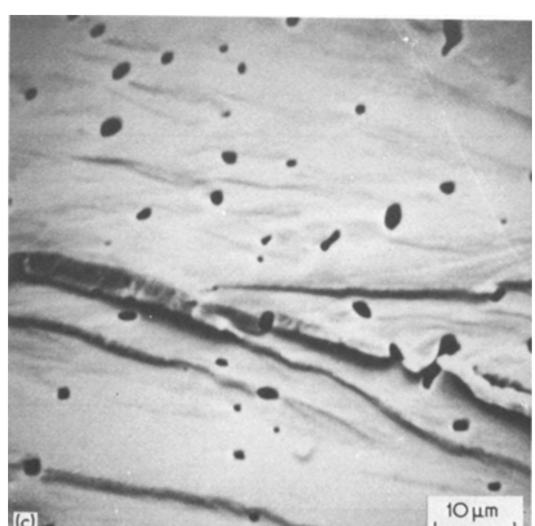


Figure 4 Scanning electron micrographs showing nature of brittle fracture (cleavage) in a tensile specimen.

TABLE I Weibull analysis for tensile test specimens

Stress, σ^i (N mm^{-2})	Failure probability $P_f^i = (i - \frac{1}{2})/N$	$X_i = \log_e \sigma^i$	$Y_i = \log_e \log_e 1/(1 - P_f^i)$
313	0.083	5.74	−2.44
434	0.250	6.07	−1.24
492	0.416	6.19	−0.618
492	0.583	6.19	−0.133
535	0.750	6.28	0.326
543	0.916	6.29	0.910[

Weibull modulus, 5.5; unit volume strength of a 1 cm cube, 356 N mm^{-2}.

TABLE II Weibull analysis for three-point bend test specimens

Stress, σ^i (N mm^{-2})	Failure probability $P_f^i = (i - \frac{1}{2})/N$	$X_i = \log_e \sigma^i$	$Y_i = \log_e \log_e 1/(1 - P_f^i)$
873	0.083	6.77	−2.44
920	0.250	6.82	−1.24
939	0.416	6.84	−0.61
951	0.583	6.85	−0.13
1198	0.750	7.08	0.32
1249	0.916	7.13	0.91

Weibull modulus, 6.9; unit volume strength of a 1 cm cube, 400 N mm^{-2}.

and that the predominant fracture mode is crystalline cleavage with a small amount of intergranular fracture. The Wallner lines which can be seen in Fig. 4 are typical of brittle cleavage in the body-centred cubic metals. A fine distribution of porosity with a pore size of 1 to 2 μm can also be seen in Fig. 4.

The six specimens which failed prematurely were tested again using a conventional brittle strength test. This was the three-point bend test described in Section 1. A span of 25 mm between the supporting knife edges was used in the experiment and a loading rate of 0.5 mm min^{-1} was adopted. Strain rates varied from zero at the supports to a maximum under the central loading knife edge. Calculations showed that the maximum strain rates for the flexural tests were comparable to those produced in the tensile test. Although the mechanical properties of sintered tungsten are sensitive to strain rate when ductile, it was felt that the slight differences in strain rate between the two test methods would not affect results in the brittle range at room temperature.

The Weibull analysis outlined in Section 2 was applied to the fracture data of the two experiments described above. The Weibull modulus and tensile failure strength of a centimetre cube were calculated for each case. These properties are, if the analysis is valid for this class of material, independent of the test method. The results of the Weibull analysis are shown in Tables I and II. Tensile and flexural tests respectively give Weibull moduli of 5.5 and 6.9, while the corresponding tensile strengths of a centimetre cube are 400 N mm^{-2} and 356 N mm^{-2}. Considering the different analytical routes involved in the evaluation of these quantities the agreement is reasonable.

Finally, the theoretical equation

$$\frac{\bar{\sigma}_{fv}}{\bar{\sigma}_{fv}^{B}} = \left[\frac{1}{2\pi^{1/2}} \frac{\Gamma\left(\frac{m+1}{2}\right)}{(m+1)\,\Gamma\left(\frac{m+4}{2}\right)}\right]^{1/m} \qquad (15)$$

relating bend strength to tensile strength was verified experimentally. A Weibull analysis based on Equation 3 was carried out on the bend test results and a quantity $\bar{\sigma}_{fv}^{B}$ evaluated. Results of this analysis are shown in Table III. Here a value of $\bar{\sigma}_{fv}^{B} = 795$ N mm^{-2} is indicated for a centimetre cube and the Weibull modulus is 6.9. Evaluating the right-hand side of Equation 15 for $m = 6.9$ gives $\bar{\sigma}_{fv}/\bar{\sigma}_{fv}^{B} = 0.503$. The experimental value of this ratio is $\bar{\sigma}_{fv}/\bar{\sigma}_{fv}^{B} = 0.448$ which again reflects reasonable agreement between the theoretical and experimental results.

TABLE III Modified Weibull analysis for three-point bend test specimens

Stress, σ^i (N mm^{-2})	Failure probability $P_f^i = (i - \frac{1}{2})/N$	$X_i = \log_e \sigma^i$	$Y_i = \log_e \log_e 1/(1 - P_f^i)$
873	0.083	6.77	−2.44
920	0.250	6.82	−1.24
939	0.416	6.84	−0.61
951	0.583	6.85	−0.13
1198	0.750	7.08	0.32
1249	0.916	7.13	0.91

Weibull modulus, 6.9; unit volume strength for a 1 cm cube, 795 N mm^{-2}.

4. Conclusion

The brittle failure characteristics of sintered tungsten have been evaluated by performing tensile and flexural tests on round bar specimens. The correlation of results for the two test methods was close considering the small samples involved. The tensile tests were more difficult to perform and are not recommended as a test procedure in the brittle range. For reliable and easier characterization it is suggested that the flexural test method is adopted.

Acknowledgements

The authors wish to thank Mrs K. Hankers for her assistance in the computation of the numerical results, Mr R. Pickering for supplying the scanning electron micrographs, and Mr D. A. R. Herrick for the mechanical testing. This paper is printed with the kind permission of the Controller, HMSO, holder of Crown Copyright.

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Received 15 January and accepted 28 March 1979.