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# THE VARIATION WITH TEMPERATURE AND POROSITY, OF THE MODULI OF RUPTURE AND ELASTICITY OF "STANDARD" ISOSTATICALLY PRESSED AND SINTERED BERYLLIA

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# THE VARIATION WITH TEMPERATURE AND POROSITY, OF THE MODULI OF RUPTURE AND ELASTICITY OF "STANDARD" ISOSTATICALLY PRESSED AND SINTERED BERYLLIA

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## ABSTRACT

Some properties of isostatically pressed and sintered UOX beryllia, fabricated at Lucas Heights to a grain size of $\leqslant 3\mu$ were measured.

The modulus of rupture of material of density 2.86 to 2.90 g $cm^{-3}$ was measured in four-point bending over the temperature range 20°C to 1000°C. The data could be represented by the equation:

$$\sigma_f = 33{,}000 - 7.46T \text{ p.s.i.}$$

The total variance was $19.5 \times 10^6$ p.s.i. made up of a "within" batch variance, of $15.8 \times 10^6$ p.s.i. and a "between" batch variance of $3.7 \times 10^6$ p.s.i.

The modulus of rupture at 20°C was measured on material with a total porosity range of 4 to 35 per cent.; the data could be represented by the equation:

$$\sigma = \sigma_0 \exp(-2.44P) \text{ p.s.i.}$$

(continued)

ABSTRACT (continued)

The modulus of elasticity was measured in four-point bending on 97.5 per cent. dense material and the data could be represented by the equation:

$$E = (51.45 \times 10^6)+(1.264 \times 10^2 T)-(1.442+10^{-1}T^2)-(2.595 \times 10^{-3}T^3) \text{ p.s.i.}$$

The modulus of elasticity was measured as a function of porosity in the range 2 to 36.4 per cent.; the data could be represented by the equation:

$$E = E_0\,(1-1.47P) \times 10^6 \text{ p.s.i.}$$

## CONTENTS

## 1. INTRODUCTION

Isostatically pressed and sintered beryllia has been adopted as the "standard" material for the coatings and matrix of fuel elements for the proposed high temperature gas-cooled reactor system which is under study by the Australian Atomic Energy Commission.

The work described below is part of a programme to evaluate the mechanical properties of the "standard" material; measurement of the moduli of elasticity and rupture as a function of temperature and porosity is reported.

## 2. MATERIAL AND SPECIMEN SIZES

A summary of the method by which the specimens of "standard" beryllia were made is given in Appendix 1. The process yielded a product which had a density of 2.86/2.90 g $cm^{-3}$; the microstructure consisted of grains having an average diameter of $\leqslant 3\mu$, with occasional grains measuring $200\mu$ in length. The large grains were associated with the presence of needle-shaped crystals of beryllia which were present in the original powder at a concentration of about 1 per cent. (Bannister 1965). The porosity was distributed mainly along grain boundaries, although some occurred in the grains themselves. Specimens that were required to have a lower density than the "standard" material were fabricated by the same method but with variations in the time and temperature of sintering. The specimens for modulus of rupture tests were machined to dimensions of 1.00 inch x 0.200 inch diameter and those for modulus of elasticity measurement to 3.5 inch x 0.400 inch x 0.125 inch.

## 3. EXPERIMENTAL METHODS

Densities were measured by a water impregnation technique and the fractional total porosity calculated from the formula:

$$P = \frac{3.01 - \rho}{3.01} ,$$

where: $P$ = fractional total porosity and

$\rho$ = density (g $cm^{-3}$).

The modulus of rupture specimens were tested in four-point bending with a gauge length of 0.31 inch and a span of 0.81 inch. In the tests at elevated temperatures, the specimens were held at temperature for 5 minutes before testing. Tests were made at 200 degree intervals in the range 20°C to 1000°C; the temperatures were accurate to ± 5°C.

The modulus of elasticity specimens were tested under static loads in four-point bending with a gauge length of 1.5 inches and a span of 3.0 inches. A sketch of the equipment used is shown in Figure 1. Basically the beryllia beam (A) is deflected in four-point bending by application of a load to the top knife edge block (B), causing the downward movement of the push rod (C);

the core (D) of a differential linear transducer (E) is attached to the end of the push rod. The transducer is attached to a tube (F) which is made from the same material as the push rod and knife edge blocks so that movement of the core in the transducer due to thermal variations is minimised.

## 4. RESULTS AND ANALYSIS

### 4.1 Modulus of Rupture versus Temperature

The results of testing 654 specimens from 19 batches (that is, different sintering runs) at six temperatures are summarised in Table 1. For any given temperature the means for each of the batches varied but this variation was not significant at the 95 per cent. confidence level; similarly the variances of the batch means were not significantly different. The data at each temperature were therefore grouped to provide a single mean and standard deviation for each temperature. The grouped means are plotted in Figure 2. A linear regression line was fitted to the data giving:

$$\sigma_f = 33{,}000 - 7.46T ,$$

where:

$\sigma_f$ = mean modulus of rupture (p.s.i.)

$T$ = temperature (°C).

This line is shown as the full line in Figure 2.

No significant differences existed between the variances of the grouped results at different temperatures, and the data at all temperatures could thus be assumed to come from the same population. The "within" batch variance was therefore estimated to be $15.8 \times 10^6$ p.s.i. Similarly, by using the regression line to provide the grand mean at any temperature, the "between" batch variance was estimated as $3.7 \times 10^6$ p.s.i. Thus the greater source of variation in the results is the "within" batch variation. The total variance (sum of within and between batch variances) was computed as $19.5 \times 10^6$ p.s.i., an effective standard deviation of 4,410 p.s.i. The 95 per cent. confidence limits about the regression line were thus calculated to be ± 8,640 p.s.i.; these confidence limits are shown as dotted lines in Figure 2.

### 4.2 Modulus of Rupture versus Fractional Total Porosity

The results of testing 161 specimens from 6 batches are shown in Table 2. If the type of equation proposed by Knudsen (1959) is assumed, where:

$$\sigma_f = \sigma_0 \, e^{-bP} ,$$

and

$\sigma_f$ = Modulus of rupture (p.s.i.)

$\sigma_0$ = Modulus of rupture at zero porosity (p.s.i.)

$P$ = Total porosity (volume fraction),

then a least mean squares analysis of log $\sigma_f$ v. P yields a value of $b = 2.44$. Because the measurement of grain sizes below 3μ is very inaccurate, no allowance has been made for grain size variation in the determination of b.

The values of this equation are plotted in Figure 3; individual results are also shown.

### 4.3 Modulus of Elasticity versus Temperature

The results from the testing of 3 specimens (density 2.94 g $cm^{-3}$) in the range 20°C to 1000°C are shown in Table 3. The means of 3 tests at each temperature are plotted in Figure 4. A regression line was fitted to the data giving:

$$E = (51.45 \times 10^6)+(1.264 \times 10^2 T)-(1.442 \times 10^{-1} T^2)-(2.595 \times 10^{-3} T^3) \text{ p.s.i.},$$

where:

$E$ = modulus of elasticity (p.s.i.)

$T$ = temperature (°C).

The standard deviation of the means from the regression line was computed to be $0.397 \times 10^6$ p.s.i. and the 95 per cent. confidence limits are $\pm 1.02 \times 10^6$ p.s.i.; these confidence limits are shown as dotted lines in Figure 4.

### 4.4 Modulus of Elasticity versus Fractional Total Porosity

The results from the testing of 12 specimens in the range 0.0216 to 0.364 fractional total porosity are shown in Table 4. The equation of the least mean squares line through the data is:

$$E = (53.06 - 78.0\,P) \times 10^6 ,$$

where:

$E$ = modulus of elasticity (p.s.i.)

$P$ = fractional total porosity.

Individual results and the regression line are shown in Figure 5. The standard deviation of the results about the regression line was computed to be $0.826 \times 10^6$ p.s.i. giving 95 per cent. confidence limits of $\pm 1.82 \times 10^6$ p.s.i. These limits are shown as dotted lines in Figure 5.

## 5. DISCUSSION

The variation of modulus of rupture of "standard" BeO with temperature is compared with extruded material (Veevers and Rotsey 1964) in Figure 6. Although the strengths were similar at 20°C and 1000°C, the "standard" material showed a continuous decrease in strength with temperature, whereas the extruded material maintained a large proportion of the room temperature strength up to 600°C.

The analysis of variance in standard material compared with that of extruded material is shown in Table 6. The greatest difference between the data lies in the "between" batch variance, the value for "standard" material being three times that of extruded material, however this variance is small compared with the "within" batch variance. The reasons for the large "within" batch variance are at present unknown and experiments are required to distinguish between variation in the material and variation due to the testing method.

The variation in modulus of rupture with porosity is similar to that observed by Collins (1963) on randomly oriented AOX beryllia. The coefficient b in the equation:

$$\sigma_f = \sigma_0 \exp(-bP) \quad ,$$

is found to be 2.44, while for AOX, Collins reported a value of 2.51. A value of 3.3 for extruded material has been determined by Kelly (A.A.E.C. unpublished); this indicates a higher degree of sensitivity to porosity than for "standard" material. The difference in the values of b is probably due to a difference in the type and distribution of porosity in the materials.

The modulus of elasticity for standard material is slightly higher than for extruded material; if the value for standard material is corrected to the same density (2.84 g cm$^{-3}$) as that of extruded material by means of the equation in Section 4.4, then the value becomes 48.0 x 10$^6$ p.s.i., whereas the equivalent figure for extruded material is 46.7 x 10$^6$ p.s.i. If failure occurs with effectively no plastic strain, then the strains to fracture can be calculated; these are shown as a function of temperature in Table 5 and plotted in Figure 7, the results for extruded material being plotted for comparison purposes. (Veevers and Rotsey, 1964). In this respect "standard" material is inferior to extruded material.

The estimated value of the modulus of elasticity for standard material of theoretical density is 53.1 x 10$^6$ p.s.i. which is similar to the value of 56.0 x 10$^6$ p.s.i. obtained from tests on AOX and UOX-$M_gO$ in both extruded and isostatically pressed condition and reported by Collins (1963). The latter measurements were made by a resonance frequency technique which generally gives slightly higher results than static loading techniques.

The variation of modulus of elasticity with total fractional porosity P is described by the equation:

$$E = 53.1\ (1 - 1.47P) \times 10^6 \text{ p.s.i.}$$

This differs from the General Electric results (Collins 1963) which are described by the equation:

$$E = 56.0\ (1 - 1.87P) \times 10^6 \text{ p.s.i.}$$

The difference in behaviour is probably associated with the distribution of porosity; unfortunately there are not sufficient data on the G.E. material to enable a comparison to be made. In the isostatically pressed material at high densities the porosity is

mainly uniformly dispersed and intragranular, changing to intergranular at high porosities with aggregates of pores being present.

## 6. CONCLUSIONS

Tests of "standard" beryllia show that:

(1) The modulus of rupture versus temperature relationship is represented by the equation:

$$\sigma = 33{,}000 - 7.46T \text{ p.s.i.}$$

The total variance is computed to be 19.5 x 10$^6$ p.s.i. consisting of a "between" batch variance of 3.7 x 10$^6$ p.s.i. and a "within" batch variance of 15.8 x 10$^6$ p.s.i.

(2) The modulus of rupture versus porosity relationship is represented by the equation:

$$\sigma = \sigma_0 \exp(-2.44P) \ .$$

(3) The modulus of elasticity versus temperature relationship for material with a density of 2.94 g cm$^{-3}$ is represented by the equation:

$$E = (51.45\text{x}10^6) + (1.264\text{x}10^2T) - (1.442\text{x}10^{-1}T^2) + (2.595\text{x}10^{-3}T^3).$$

(4) The modulus of elasticity versus porosity relationship is represented by the equation:

$$E = (53.1 - 78.0\,P) \times 10^6 \text{ p.s.i.}$$

## 7. ACKNOWLEDGEMENTS

Much of the work was done by K. J. Ireland and the specimens were supplied by members of the Ceramics Group.

## 8. REFERENCES

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Collins, C.G. (1963). - 2nd Annual Report. High Temperature Materials and Reactor Component Development Programs. February 28 1963 GEMP 177A.

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## TABLE 1

## VARIATION IN MEAN MODULUS OF RUPTURE WITH TEMPERATURE

| Temperature °C | No. of Tests | Mean Modulus of Rupture p.s.i. | Standard Deviation p.s.i. | 95% Confidence Limits p.s.i. | Coefficient of Variation % |
|---|---|---|---|---|---|
| 20 | 253 | 32,800 | 5,000 | ± 9,800 | 15.25 |
| 200 | 89 | 32,100 | 4,890 | ± 9,780 | 15.25 |
| 400 | 85 | 29,000 | 3,970 | ± 7,940 | 13.80 |
| 600 | 86 | 28,500 | 4,590 | ± 9,180 | 16.15 |
| 800 | 86 | 28,300 | 5,020 | ± 10,040 | 17.65 |
| 1000 | 55 | 24,900 | 5,040 | ± 10,080 | 20 |

Total Number of Specimens - 564

Number of Batches - 19

## TABLE 2

## MODULUS OF RUPTURE VERSUS FRACTIONAL TOTAL POROSITY

| Fractional Porosity | Modulus of Rupture p.s.i. | Fractional Porosity | Modulus of Rupture p.s.i. |
|---|---|---|---|
| 0.0365 | 27,700 | 0.0332 | 39,100 |
| 0.0399 | 27,700 | 0.0432 | 28,500 |
| 0.0399 | 29,000 | 0.0498 | 38,800 |
| 0.0432 | 28,100 | 0.0498 | 34,600 |
| 0.0498 | 24,700 | 0.0498 | 22,500 |
| 0.0332 | 32,800 | 0.0498 | 33,700 |
| 0.0332 | 30,300 | 0.0565 | 32,300 |
| 0.0365 | 33,100 | 0.0399 | 35,800 |
| 0.0399 | 32,300 | 0.0498 | 38,200 |
| 0.0365 | 31,700 | 0.0532 | 29,800 |
| 0.0332 | 34,000 | 0.0465 | 36,600 |
| 0.0399 | 38,100 | 0.0930 | 33,700 |
| 0.0598 | 37,600 | 0.0764 | 30,700 |
| 0.0399 | 39,600 | 0.0831 | 29,900 |
| 0.0299 | 39,700 | 0.0897 | 33,000 |
| 0.0399 | 40,100 | 0.0698 | 27,300 |
| 0.0532 | 36,100 | 0.0697 | 33,600 |
| 0.0465 | 33,400 | 0.0731 | 29,000 |
| 0.0365 | 39,100 | 0.0631 | 32,100 |
| 0.0365 | 36,900 | 0.0731 | 26,300 |
| 0.0399 | 38,000 | 0.0764 | 37,500 |
| 0.0332 | 37,000 | 0.0665 | 34,400 |
| 0.0465 | 37,800 | 0.0867 | 30,800 |
| 0.0432 | 39,100 | 0.0651 | 28,200 |
| 0.0432 | 44,400 | 0.0837 | 28,300 |
| 0.0598 | 38,900 | 0.1033 | 15,400 |
| 0.0365 | 37,700 | 0.0581 | 31,500 |
| 0.0332 | 32,700 | 0.0774 | 24,600 |
| 0.0332 | 39,000 | 0.0751 | 32,100 |
| 0.0299 | 33,600 | 0.0857 | 29,300 |
| 0.0465 | 33,700 | 0.0841 | 23,400 |
| 0.0399 | 43,100 | 0.0789 | 30,200 |
| 0.0465 | 38,300 | 0.0508 | 27,800 |
| 0.0432 | 39,400 | 0.0588 | 17,500 |
| 0.0432 | 34,900 | 0.0890 | 26,500 |
| 0.0432 | 41,200 | 0.0223 | 32,700 |
| 0.0465 | 43,300 | 0.0518 | 30,100 |
| 0.0432 | 41,800 | 0.0841 | 26,300 |
| 0.0432 | 40,600 | 0.0837 | 32,100 |
| 0.0465 | 34,300 | 0.1076 | 22,600 |
| 0.0432 | 39,500 | 0.0950 | 28,900 |
| 0.0432 | 35,800 | 0.0804 | 30,800 |
| 0.0399 | 38,600 | 0.1588 | 26,700 |
| 0.0432 | 28,100 | 0.1681 | 25,300 |
| 0.0432 | 40,600 | 0.1714 | 25,400 |
| 0.0432 | 30,600 | 0.1618 | 25,400 |
| 0.0465 | 33,800 | 0.1658 | 25,200 |
| 0.0432 | 34,800 | 0.1575 | 23,200 |
| 0.0432 | 36,400 | 0.1545 | 29,400 |
| 0.0399 | 34,200 | 0.1565 | 31,200 |

(continued)

## TABLE 2 (continued)

| Fractional Porosity | Modulus of Rupture p.s.i. | Fractional Porosity | Modulus of Rupture p.s.i. |
|---|---|---|---|
| 0.1651 | 27,600 | 0.2498 | 23,400 |
| 0.1532 | 26,100 | 0.2236 | 22,700 |
| 0.0920 | 21,500 | 0.2588 | 18,700 |
| 0.1518 | 27,400 | 0.2302 | 23,800 |
| 0.1654 | 31,400 | 0.2741 | 18,700 |
| 0.1492 | 23,400 | 0.2741 | 21,700 |
| 0.1595 | 29,900 | 0.3186 | 17,000 |
| 0.1601 | 28,500 | 0.3243 | 16,300 |
| 0.1701 | 22,700 | 0.2608 | 25,400 |
| 0.1681 | 23,600 | 0.2372 | 26,200 |
| 0.1661 | 31,800 | 0.3502 | 16,800 |
| 0.1661 | 26,200 | 0.3399 | 15,700 |
| 0.1698 | 18,700 | 0.3621 | 18,400 |
| 0.1565 | 30,700 | 0.3542 | 18,100 |
| 0.1498 | 25,200 | 0.3691 | 18,600 |
| 0.1495 | 30,800 | 0.3419 | 19,400 |
| 0.2648 | 16,700 | 0.3661 | 14,500 |
| 0.2548 | 18,100 | 0.3674 | 16,400 |
| 0.2887 | 25,400 | 0.3625 | 10,400 |
| 0.2515 | 22,200 | 0.3701 | 10,400 |
| 0.2505 | 23,100 | 0.3811 | 16,900 |
| 0.2488 | 19,700 | 0.3844 | 17,700 |
| 0.2728 | 19,400 | 0.3555 | 21,300 |
| 0.2508 | 20,000 | 0.3744 | 9,700 |
| 0.2934 | 21,400 | 0.3824 | 17,400 |
| 0.3538 | 20,400 | 0.3910 | 13,800 |
| 0.2326 | 20,100 | 0.3754 | 12,400 |
| 0.3532 | 22,500 | 0.3618 | 17,000 |
| 0.2628 | 19,600 | 0.3495 | 14,600 |
| 0.2767 | 12,300 | | |
| 0.2532 | 17,400 | | |

## TABLE 3

## MODULUS OF ELASTICITY VERSUS TEMPERATURE FOR SPECIMENS OF DENSITY 2.94 g cm$^{-3}$

| Temperature °C | Modulus of Elasticity ($10^6$ p.s.i.) 1 | 2 | 3 | Mean |
|---|---|---|---|---|
| 20 | 52.20 | 50.60 | 51.60 | 51.46 |
| 200 | 51.90 | 51.00 | 51.40 | 51.43 |
| 400 | 51.80 | 50.90 | 51.10 | 51.26 |
| 600 | 51.50 | 50.20 | 51.40 | 51.03 |
| 800 | 49.90 | 49.70 | 50.80 | 50.13 |
| 1000 | 49.20 | 48.40 | 49.20 | 48.86 |

## TABLE 4

## MODULUS OF ELASTICITY VERSUS FRACTIONAL TOTAL POROSITY

| Fractional Porosity | Modulus of Elasticity ($10^6$ p.s.i.) |
|---|---|
| 0.0250 | 51.60 |
| 0.0233 | 50.10 |
| 0.0216 | 51.10 |
| 0.0848 | 47.00 |
| 0.1810 | 39.35 |
| 0.1810 | 40.20 |
| 0.2780 | 32.10 |
| 0.2625 | 32.25 |
| 0.2710 | 31.05 |
| 0.3490 | 25.70 |
| 0.3460 | 24.90 |
| 0.3640 | 25.75 |

## TABLE 5

## CALCULATED STRAIN AT FRACTURE VERSUS TEMPERATURE

| Temperature °C | E (10⁶ p.s.i.) | $\sigma_f$ p.s.i. | $\varepsilon$ x $10^{-4}$ | Material |
|---|---|---|---|---|
| 20 | 48.00 | 32,900 | 6.85 | Isostatically Pressed BeO |
| 200 | 47.80 | 31,500 | 6.59 | |
| 400 | 47.68 | 30,000 | 6.29 | |
| 600 | 47.45 | 28,600 | 6.04 | |
| 800 | 46.65 | 27,100 | 5.82 | |
| 1000 | 45.50 | 25,600 | 5.63 | |
| 20 | 46.60 | 33,300 | 7.15 | Extruded and Sintered BeO |
| 200 | 46.61 | 33,100 | 7.19 | |
| 400 | 46.25 | 32,400 | 7.01 | |
| 600 | 44.94 | 31,150 | 6.93 | |
| 800 | 42.00 | 28,800 | 6.86 | |
| 1000 | 41.00 | 25,500 | 6.23 | |

## TABLE 6

## ANALYSIS OF VARIANCE FOR "STANDARD" AND EXTRUDED MATERIALS

| | "Standard" Material | Extruded Material |
|---|---|---|
| Total Variance ($10^6$ p.s.i.) | 19.5 | 17.9 |
| "Between" Batch Variance ($10^6$ p.s.i.) | 3.7 | 1.2 |
| "Within" Batch Variance ($10^6$ p.s.i.) | 15.8 | 16.7 |

## APPENDIX 1

## SUMMARY OF THE FABRICATION ROUTE FOR "STANDARD" BERYLLIA

(1) The powder is homogenised by grinding for one hour in water with beryllia balls followed by sieving, filtering, and drying.

(2) The dried powder is pre-formed in a steel die at 0.25 to 0.50 tons per square inch.

(3) The pre-formed sample is then isostatically pressed in a rubber envelope at 20 tons per square inch.

(4) The sample is then sintered in dry nitrogen for 75 minutes at 1500°C.

(5) The sample is centreless ground to size with SiC wheels and then annealed for 4 hours at 800°C.

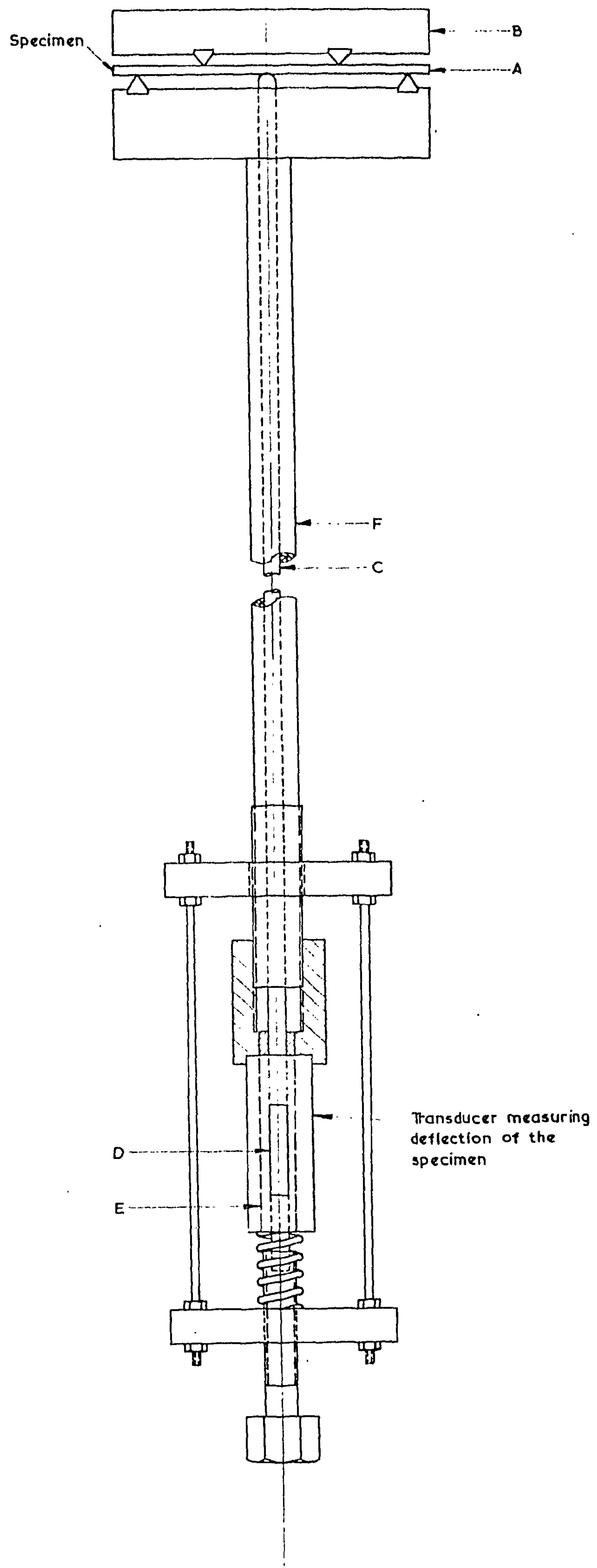

FIGURE 1 YOUNG'S MODULUS APPARATUS

P662

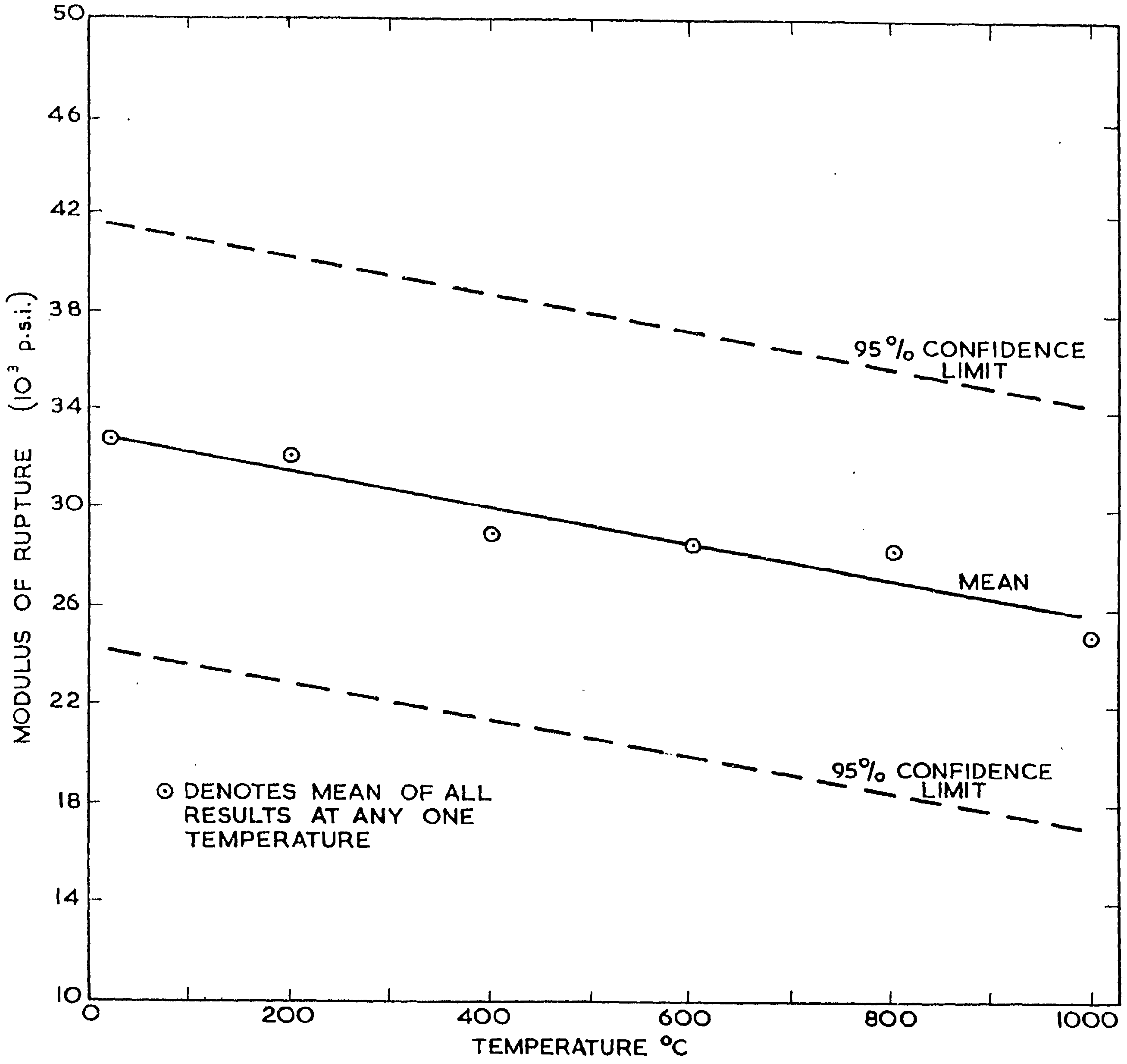

FIGURE 2. MODULUS OF RUPTURE V. TEMPERATURE

P743

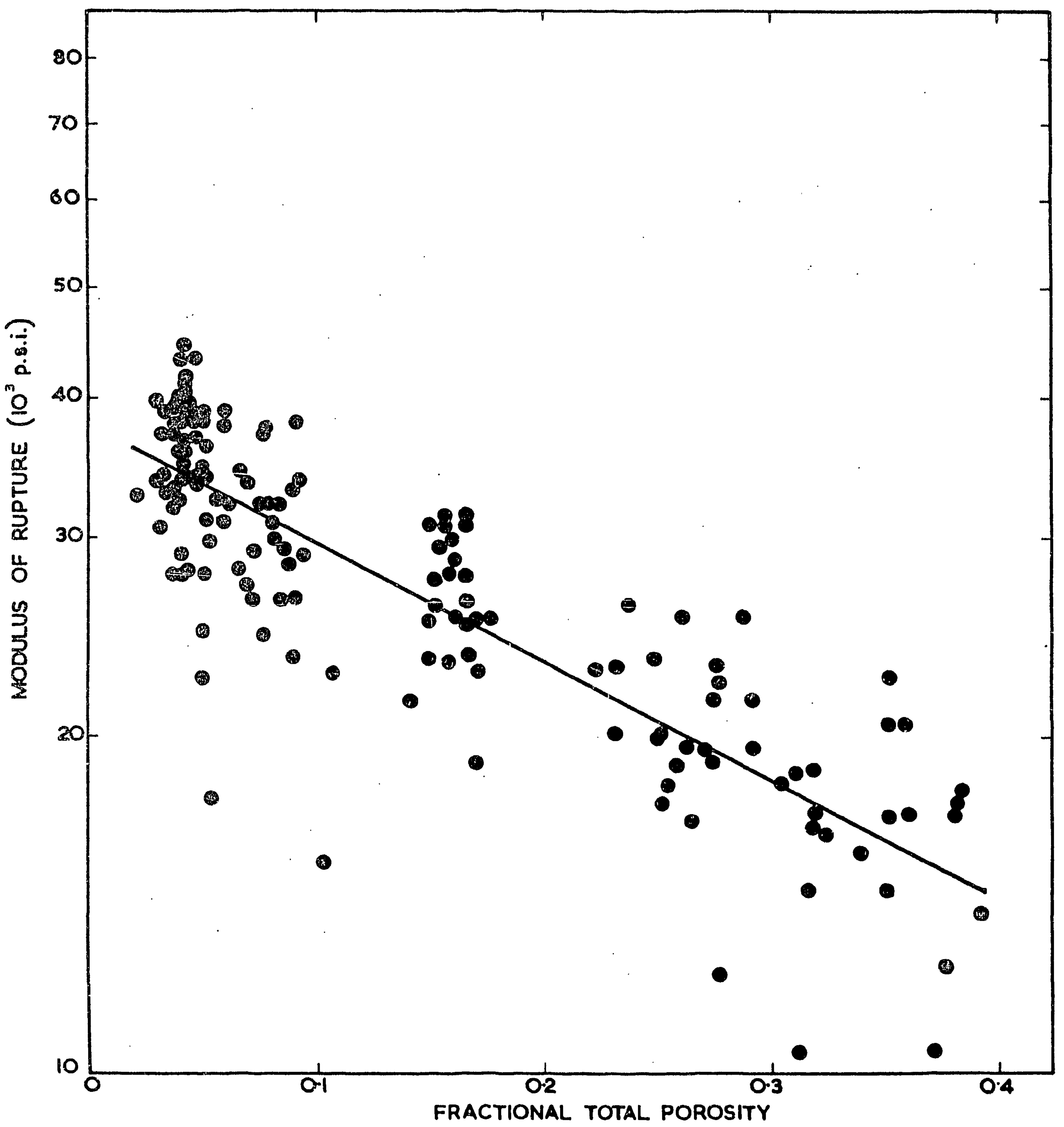

FIGURE 3. MODULUS OF RUPTURE V. FRACTIONAL TOTAL POROSITY

P743

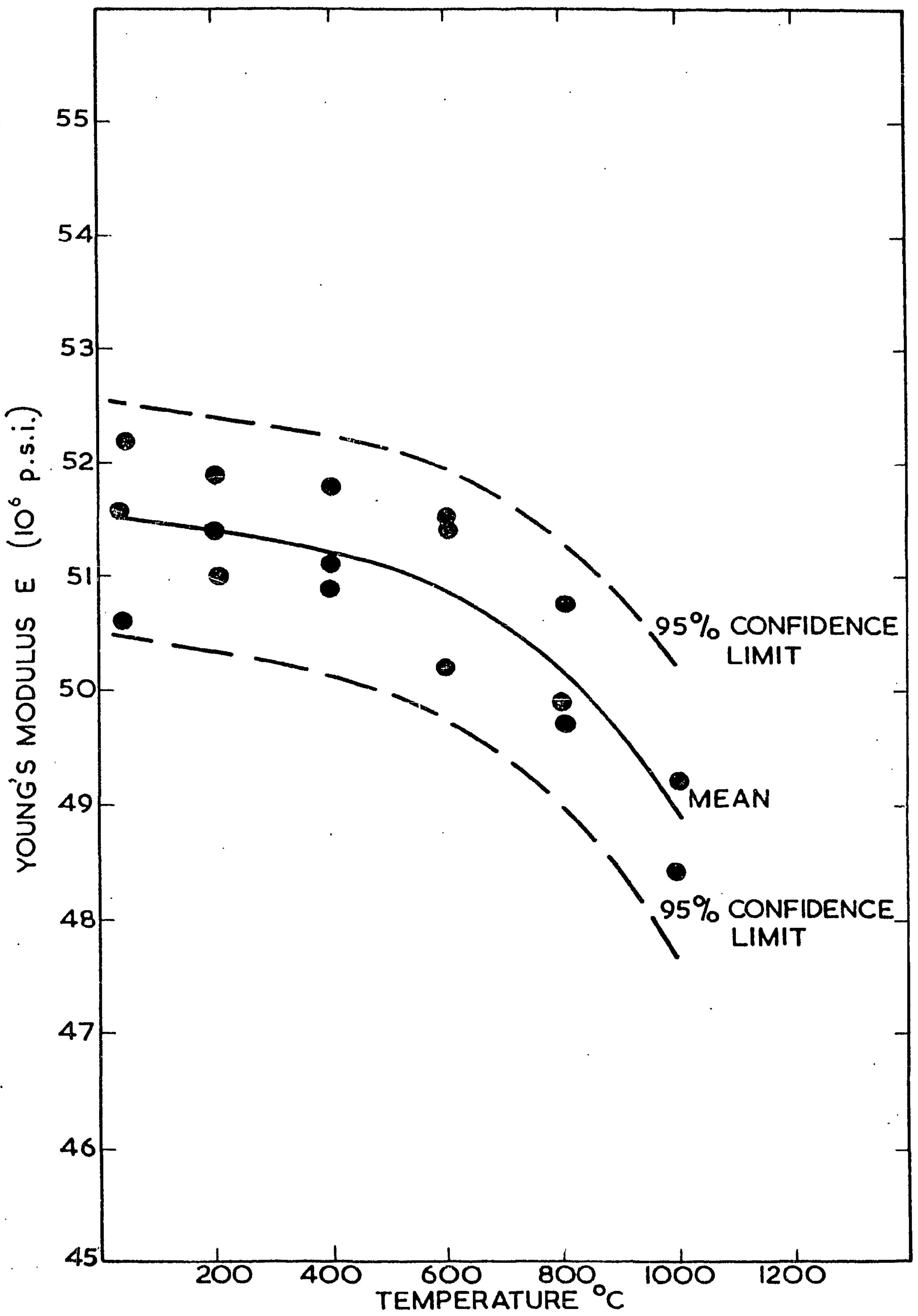

FIGURE 4. YOUNG'S MODULUS OF "STANDARD" BeO V. TEMPERATURE

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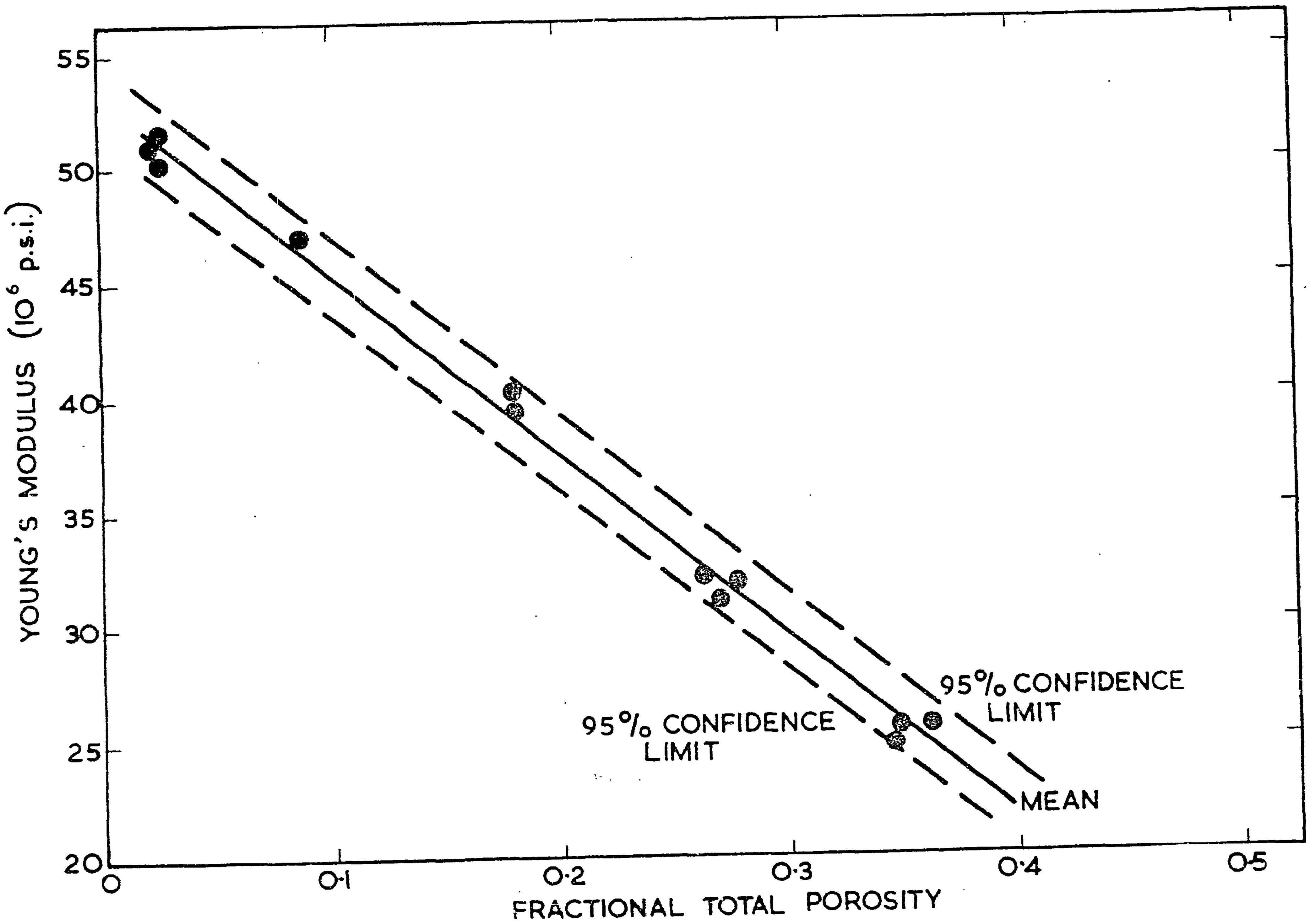

FIGURE 5. YOUNG'S MODULUS V. FRACTIONAL TOTAL POROSITY

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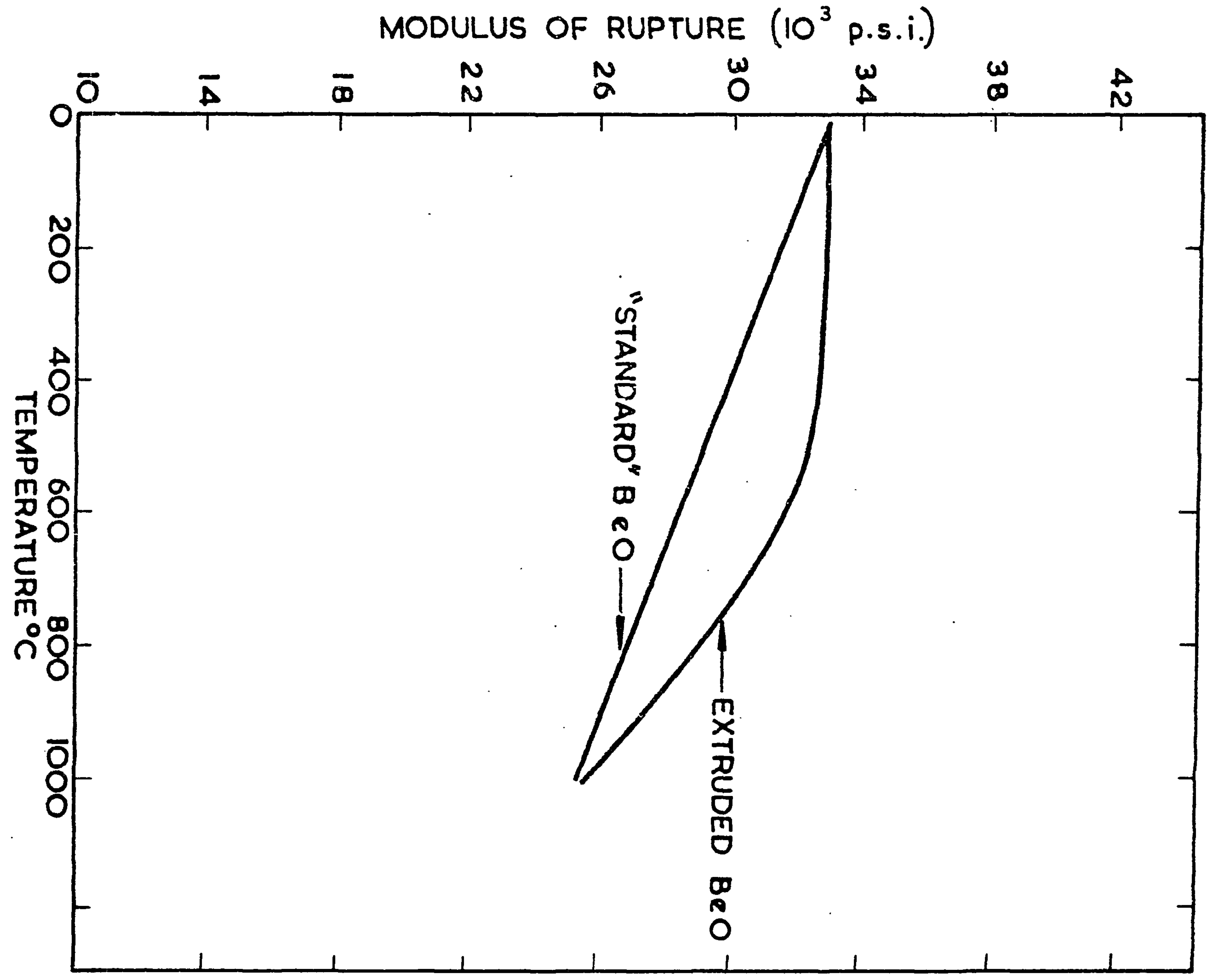

FIGURE 6. COMPARISON OF EXTRUDED AND SINTERED BeO WITH "STANDARD" BeO V. TEMPERATURE

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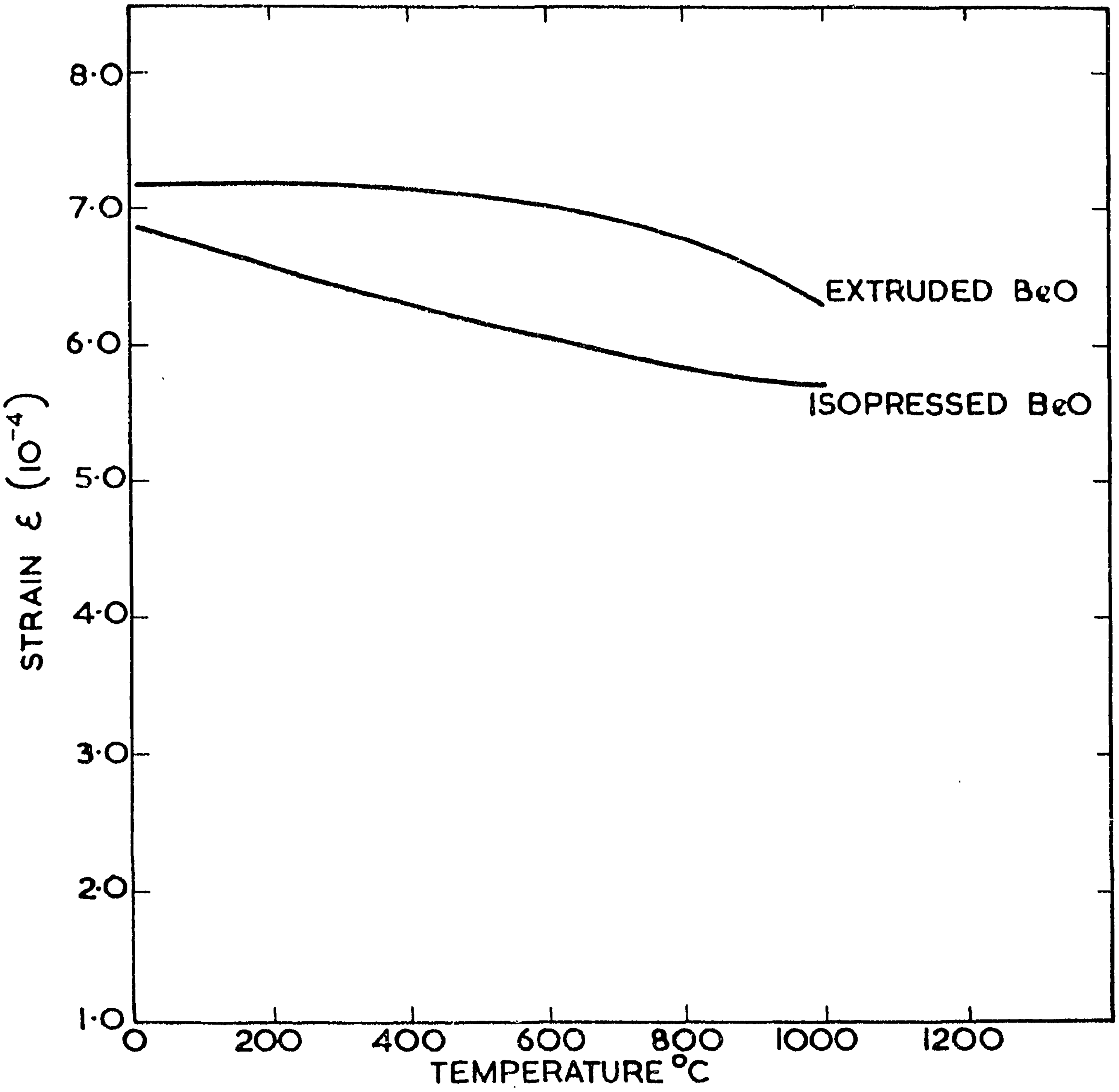

FIGURE 7. CALCULATED STRAIN TO FRACTURE V. TEMPERATURE