NASA CR-66933

18
ILB

ELASTIC PROPERTIES OF THREE GRADES OF FINE GRAINED GRAPHITE TO 2000°C

M. O. MARLOWE

NASA Contract NAS1-9852
June 25, 1970

NUCLEONICS LABORATORY • GENERAL ELECTRIC COMPANY
VALLECITOS NUCLEAR CENTER, PLEASANTON, CALIFORNIA, 94566

GENERAL ELECTRIC

NASA CR–66933

ELASTIC PROPERTIES OF THREE GRADES OF FINE GRAINED GRAPHITE TO 2000^{o}C

M. O. Marlowe

NASA Contract NAS1–9852
June 25, 1970

NUCLEONICS LABORATORY ● GENERAL ELECTRIC COMPANY
VALLECITOS NUCLEAR CENTER, PLEASANTON, CALIFORNIA, 94566

NEDG-12106
3290-NL-34
75-DAC-10/70

GENERAL ELECTRIC

LEGAL NOTICE

Requests for copies of this report should be referred to:

National Aeronautics and Space Administration
Scientific and Technical Information Facility
P. O. Box 33
College Park, Maryland 20740

CONTENTS

ELASTIC PROPERTIES OF THREE GRADES OF FINE GRAINED GRAPHITE TO 2000°C

M. O. Marlowe

ABSTRACT

The room temperature and elevated temperature (to 2000°C) Young's modulus and shear modulus of three grades of fine-grained graphite were determined. The materials tested were ATJS, POCO 5Q and POCO 9Q. The room temperature moduli were found to be directly proportional to the density of the specimens. However, each grade was characterized by a different density dependence and extrapolated moduli values at full density. The moduli of each grade increased with temperature, the initial rate of increase increasing in the order POCO 5Q, POCO 90 and ATJS. The moduli reached maximum values in the temperature ranges 1250 to 1550°C for the POCO 5Q, and 1700 to 1750°C for the POCO 9Q but no maxima were achieved below 2000°C for the ATJS. A hysteresis of up to 500°C was observed in the moduli on cooling. There was considerable spread in the temperature dependence of the elasticity of the samples from a given graphite grade.

INTRODUCTION

The mechanical design and assessment of the thermal shock behavior of rocket nozzle materials requires an accurate knowledge of the elasticity of the candidate materials. It is the purpose of this report to present room temperature and elevated temperature (to approximately 2000°C) elasticity data on three grades of graphite, *i.e.,* ATJS, POCO 5Q and POCO 9Q.

MATERIALS AND SPECIMENS

Specimens of three grades of graphite were received from NASA–Langley for determination of their room temperature and elevated temperature elasticity. The graphite grades were ATJS manufactured by Union Carbide Corporation, New York, N. Y. and POCO 5Q and POCO 9Q manufactured by Poco Graphite, Inc., Garland, Texas.

Two types of specimen geometry were used. They were rectangular bar shaped specimens approximately 0.35 in. X 0.45 in. X 5.0 in., and cylindrical specimens approximately 0.3 in. in diameter and 3.7 in. long. The rectangular specimens were used for the detailed room temperature elasticity measurements and the cylindrical specimens were used for determination of the temperature dependence of elasticity of the graphites.

The weight, dimensions and density of the specimens are summarized in Table 1.

MEASUREMENT TECHNIQUES

Young's and shear moduli of elasticity were determined by the sonic resonant frequency technique.[1] The room temperature resonant frequencies and their modes of vibration were unequivocally determined by probing as suggested by Spinner and Tefft.[2] The fundamental flexural, torsional and longitudinal resonant frequencies were determined at room temperature by exciting the specimen with a speaker (Electro–Voice, Inc., Model TW–35 VHF Driver) and directly contacting the specimen with the pickup (Astatic Type 320). Elevated temperature data were obtained in vacuum at less than 4×10^{-4} torr.

For those data, the specimens were suspended in a resistance heated tungsten furnace on Pluton* strings. The suspension strings were glued to the driver rod (Astatic Type M41–8 driver) and the pickup beam from the pickup (Astatic Type 445). A ceramic type pickup cartridge was chosen to avoid deterioration of the pickup in vacuum. Fundamental flexural and torsional resonant frequencies were determined at temperature.

---

*"Pluton" Type 10A, 8 ounce per square yard fabric obtained from Minnesota Mining and Manufacturing Co. was used as the suspension strings.

TABLE 1. SPECIMEN CHARACTERIZATION

Specimen Number	Thickness or Diameter (in.)	Width (in.)	Length (in.)	Weight (gm)	Density (% of Theoretical)[1]
POCO 5Q-1	0.351[3]	0.451[3]	5.004	23.6151	80.7319
POCO 5Q-2	0.351	0.451	5.003	23.4773	80.3455
POCO 5Q-3	0.351[6]	0.451	5.003	23.3965	79.9855
POCO 5Q-4	0.351	0.451	5.002	23.3986	80.1454
POCO 5Q-5	0.351	0.451	5.003	23.4505	80.3072
POCO 5Q-6	0.350	0.450[3]	4.983[5]	23.2505	80.2868
POCO 5Q-7	0.313[2]	-	4.059	9.0263	78.2875
POCO 5Q-8	0.313[3]	-	3.739	8.8551	83.3245
POCO 5Q-9	0.312	-	3.696	8.0948	77.6979
POCO 9Q-1	0.351	0.451	5.003	23.7708	81.4040
POCO 9Q-2	0.351	0.451	5.005	23.7103	81.1644
POCO 9Q-2[2]	0.169[3]	-	3.535	2.3923	81.5341
POCO 9Q-3	0.351[3]	0.451	5.005	23.7233	81.1396
POCO 9Q-4	0.351	0.451	5.003	23.7971	81.4941
POCO 9Q-5	0.351	0.451[3]	5.004	23.5994	80.7472
POCO 9Q-6	0.351	0.451	5.001	23.8130	81.5115
POCO 9Q-7	0.178	-	3.753	2.7906	81.0511
POCO 9Q-8	0.313[1]	-	3.741	8.6275	81.2390
ATJS-1	0.351[3]	0.452	5.003[5]	24.0234	82.0088
ATJS-2	0.351[3]	0.452	5.006	24.0796	82.1596
ATJS-3	0.351[3]	0.451[6]	5.003	23.2582	79.4749
ATJS-4	0.351[3]	0.451[6]	5.005	23.9799	81.9082
ATJS-5	0.351	0.452	5.003	23.5510	80.4729
ATJS-6	0.351	0.451[6]	5.004	24.2526	80.2868
ATJS-8	0.321[9]	-	3.690	9.0752	81.9700
ATJS-9	0.321[8]	-	3.695	9.0823	81.9705
ATJS-10	0.321[8]	-	3.621	9.1835	84.5777

(1) Percent of theoretical density based on 2.25 gm/cm^3 as the theoretical density of graphite.

(2) Machined from POCO 9Q-2.

For the room temperature data, ten determination of the resonant frequencies were made for each specimen in each mode of vibration. The frequencies were determined with a frequency counter (Transistor Specialties, Inc., Model 361–R) using a 10–second count when the spread between individual readings would warrant it. Otherwise, 1–second counts were obtained.

Elevated temperature data were obtained under isothermal conditions after maintaining the measurement temperature for a sufficient time that there was no longer a detectable drift in the resonant frequencies with time. Specimen temperature was sensed with a 95% tungsten/5% rhenium/74% tungsten/26% rhenium thermocouple in a thoria tube located at the center of the specimen and adjacent to it.

The ratio of the elastic moduli at elevated temperatures M_T to that at room temperature M_O were calculated using the relation:

$$M_T/M_O = (f^2_{M_T} / f^2_{M_O}) / [(1 + \Delta L_T / L_O)] \tag{1}$$

where f_{M_T} and f_{M_O} are the appropriate resonant frequencies for the particular modulus of interest (Young's or shear) at the corresponding temperature and $\Delta L_T/L_O$ is the fractional linear thermal expansion of the material on changing temperature from room temperature to the temperature of interest. The thermal expansion of ATJS graphite was approximated by the relation:

$$\Delta L_T/L_O = -9.85 \times 10^{-5} + 3.94 \times 10^{-6}\, T \tag{2}$$

where T is the temperature in $^{\circ}$C. The expansion given by this relationship falls approximately midway between the thermal expansion data for ATJS graphite in the grain and across the grain directions[3] for the temperature range of interest (to 2000°C). The thermal expansion of both grades of POCO graphite was approximated by the relation:

$$\Delta L_T/L_O = 1.155 \times 10^{-4} + 6.513 \times 10^{-6}\, T + 9.25 \times 10^{-10}\, T^2 \tag{3}$$

where T is the temperature in $^{\circ}$C. The expansion given by this relationship falls midway between the thermal expansion data[4] for POCO 5Q in the temperature range of interest. No additional expansion data were found for the POCO 9Q material.

Qualitative assessment of the internal friction of the specimens as a function of temperature was made by comparing the amplitude of the pickup output at constant input voltage to the driver.

RESULTS

ROOM TEMPERATURE ELASTICITY

The room temperature resonant frequencies and their standard deviations for the prismatic bar specimens are listed in Table 2. The two values of the torsional resonant frequencies were used to calculate an average value for the room temperature shear modulus G, from the relation:

$$G = \rho\, (2Lf_T)^2\, R \tag{4}$$

where ρ is the sample density, L is its length and f_T is the fundamental torsional resonant frequency and R is a shape factor for prisms of rectangular cross section as given by Spinner and Tefft.[2]

The relations:

$$E = (0.94642\, \rho\, L^4\, f_F^2\, T)\, t^2 \tag{5}$$

and

$$E = (\rho / K)\, (2L\, f_L)^2$$

TABLE 2. ROOM TEMPERATURE RESONANT FREQUENCY RESULTS

Specimen Number	Fundamental Resonant Frequencies*				
	Flexure		Torsion		
	Flatwise	Edgewise	Flatwise	Edgewise	Longitudinal
POCO 5Q-1	1478.6 ± 0.1	1874.8 ± 0.1	5819.4 ± 0.7	5818.1 ± 0.7	10407.8 ± 0.5
POCO 5Q-2	1495.1 ± 0.1	1899.8 ± 0.1	5869.0 ± 0.7	5877.8 ± 0.6	10531.6 ± 0.6
POCO 5Q-3	1471.3 ± 0.04	1869.3 ± 0.1	5804.0 ± 1.1	5804.9 ± 0.6	10364.1 ± 0.3
POCO 5Q-4	1480.3 ± 0.0	1877.3 ± 0.1	5821.1 ± 0.6	5823.1 ± 1.0	10403.2 ± 0.5
POCO 5Q-5	1496.0 ± 0.1	1898.4 ± 0.1	5868.6 ± 0.8	5875.6 ± 0.7	10515.3 ± 0.4
POCO 5Q-6	1494.4 ± 0.1	1901.2 ± 0.1	5873.6 ± 1.3	5883.8 ± 0.6	10521.7 ± 0.5
POCO 9Q-1	1432.2 ± 0.1	1816.6 ± 0.2	5636.7 ± 0.7	5638.5 ± 0.8	10058.0 ± 0.7
POCO 9Q-2	1385.2 ± 0.04	1759.4 ± 0.1	5521.8 ± 1.3	5527.4 ± 0.7	9746.9 ± 0.7
POCO 9Q-3	1363.6 ± 0.1	1725.1 ± 0.1	5441.8 ± 1.2	5441.8 ± 0.9	9574.1 ± 0.7
POCO 9Q-4	1403.3 ± 0.1	1783.9 ± 0.1	5563.4 ± 1.2	5561.4 ± 1.1	9885.4 ± 0.7
POCO 9Q-5	1347.8 ± 0.1	1708.0 ± 0.1	5392.3 ± 1.2	5390.7 ± 0.9	9475.2 ± 0.8
POCO 9Q-6	1425.5 ± 0.1	1806.6 ± 0.1	5624.8 ± 0.9	5624.7 ± 0.5	10011.4 ± 0.5
ATJS-1	1417.8 ± 0.1	1810.7 ± 0.2	5847.0 ± 1.1	5838.4 ± 1.3	10013.2 ± 1.4
ATJS-2	1451.0 ± 0.1	1839.9 ± 0.2	5788.8 ± 1.0	5787.9 ± 1.2	10200.2 ± 0.4
ATJS-3	1393.3 ± 0.1	1770.2 ± 0.1	5666.8 ± 0.4	5666.0 ± 0.9	9769.1 ± 0.7
ATJS-4	1437.1 ± 0.1	1824.0 ± 0.1	5772.8 ± 0.9	5769.3 ± 1.3	10095.2 ± 0.4
ATJS-5	1419.9 ± 0.1	1804.3 ± 0.1	5725.9 ± 0.9	5720.7 ± 0.9	9985.2 ± 0.4
ATJS-6	1435.7 ± 0.1	1822.2 ± 0.1	5745.5 ± 1.2	5742.6 ± 0.5	10100.0 ± 0.8

* The ± values are one standard deviation.

where t is the thickness of the specimen in the plane of flexure, f_F is the appropriate flexural resonant frequency, and f_L is the fundamental longitudinal resonant frequency were used to calculate values of the Young's modulus E. Here T and K are correction factors that depend on the Poisson's ratio and the thickness and length of the specimens.

By assuming that the specimens were elastically isotropic, and hence the Poisson's ratio, μ, is given by the following simple function of the Young's modulus and the shear modulus:

$$\mu \; (2E/G) - 1 \tag{6}$$

the room temperature data allowed four values of the Young's modulus to be calculated. These four values were obtained as follows. A reasonable value for the Poisson's ratio was assumed and the correction factor T was calculated for the flatwise flexural mode of vibration. This correction factor and the dimensions and resonant frequency were then used to calculate a value of the Young's modulus using Equation (5). The Poisson's ratio was then calculated using Equation (6), and the new value of Poisson's ratio was used in a recalculation of the correction factor T. The above steps were then repeated until a consistent set of values for the Young's modulus and the Poisson's ratio was obtained to the accuracy desired (3 decimal places). Having obtained a value for the Poisson's ratio, that value was used for calculation of the correction factor K, and a calculation of the Young's modulus from the longitudinal resonant frequency. The above process was repeated then using the edgewise flexural resonant frequency, thus yielding four values of the Young's modulus and two values of the Poisson's ratio. The four computed values of Young's modulus; the two values of Poisson's ratio and the average shear modulus value noted above were used to prepare the summary of room temperature elastic moduli values listed in Table 3.

These room temperature moduli values are plotted as a function of density, ρ, ((% of theoretical) in Figures 1–3. The solid lines are least squares fits of the experimental data by the relationship:

$$M = Mo \; (1 - BP)$$

where Mo and B are constants and P is the volume fraction porosity (*i.e.,* $P = \rho/100$). The extrapolated moduli at full density, Mo and the moduli versus porosity slopes, B are listed in Table 4.

ELEVATED TEMPERATURE ELASTICITY AND INTERNAL FRICTION

The ratio of the elevated temperature Young's and shear moduli to the room temperature values are plotted in Figures 4 to 16. Both heating and cooling were obtained, except on Specimen POCO 5Q–7. The heating data are summarized in Figure 17 and 18.

The results of qualitative observations of the internal friction of the specimens as a function of temperature were as follows. For all specimens, there was a decrease in damping with increasing temperature on heating to approximately half the room temperature value, followed by a rise in internal friction to approximately twice that at room temperature at the maximum temperature of measurement (2000°C). The transition from decreasing to increasing internal friction with temperature, *i.e.,* the internal friction minima, varied with the graphite grade as listed in Table 5.

DISCUSSION

ROOM TEMPERATURE ELASTICITY

As shown in Figures 1 to 3, there is a good correlation of the room temperature elastic moduli with density. However, apparent steep slopes of the moduli of the POCO 9Q graphite with density leads one to question the statistical validity of the least squares fitted lines for the purposes of extrapolation. More data on more specimens and with a greater range of densities is clearly needed to better define the exact relationship between the graphite density and porosity. This again is especially evident in the case of the POCO 90 graphite in examining the effect of density on the Poisson's ratio. The extrapolation to fully dense material gives impossible values for the Poisson's ratio of the fully dense material. These results do indicate the need for considering the effect of density on the elastic moduli of these materials, as the normal ~ 2.5% spread or less in density in the specimens was reflected in moduli differences up to 12.5%.

TABLE 3. SUMMARY OF ROOM TEMPERATURE ELASTICITY RESULTS*

Specimen Number	Young's Modulus (10^6 psi)	Shear Modulus (10^6 psi)	Poisson's Ratio
POCO 5Q-1	1.844 ± 0.004	0.724	0.273 ± 0.003
POCO 5Q-2	1.880 ± 0.001	0.734 ± 0.002	0.280 ± 0.001
POCO 5Q-3	1.811 ± 0.003	0.713	0.269 ± 0.002
POCO 5Q-4	1.831 ± 0.004	0.719	0.275 ± 0.003
POCO 5Q-5	1.859 ± 0.031	0.733 ± 0.001	0.258 ± 0.025
POCO 5Q-6	1.859 ± 0.001	0.729 ± 0.002	0.274
POCO 5Q-AVG.	1.847 ± 0.024	0.725 ± 0.008	0.272 ± 0.008
POCO 9Q-1	1.741 ± 0.005	0.685	0.274 ± 0.002
POCO 9Q-2	1.630 ± 0.002	0.656 ± 0.001	0.242
POCO 9Q-3	1.571 ± 0.005	0.637	0.235 ± 0.005
POCO 9Q-4	1.680 ± 0.005	0.668	0.258
POCO 9Q-5	1.530 ± 0.005	0.622	0.230 ± 0.005
POCO 9Q-6	1.723 ± 0.003	0.682	0.265 ± 0.003
POCO 9Q-AVG.	1.646 ± 0.084	0.658 ± 0.025	0.251 ± 0.018
ATJS-1	1.730 ± 0.006	0.742 ± 0.002	0.164 ± 0.004
ATJS-2	1.805 ± 0.005	0.730	0.235 ± 0.005
ATJS-3	1.604 ± 0.006	0.676	0.191 ± 0.002
ATJS-4	1.764 ± 0.004	0.723 ± 0.001	0.221 ± 0.002
ATJS-5	1.692 ± 0.004	0.699 ± 0.001	0.211 ± 0.003
ATJS-6	1.777 ± 0.004	0.722 ± 0.001	0.230 ± 0.003
ATJS-AVG.	1.729 ± 0.073	0.715 ± 0.024	0.209 ± 0.027

*The ± values shown are <u>one</u> standard deviation; if none is shown the standard deviation was less than one in the third decimal phase for the listed value.

TABLE 4. EXTRAPOLATED ROOM TEMPERATURE ELASTIC MODULI AT 100% DENSITY

Graphite Grade	Extrapolated 100% Density Young's Modulus (10^6 psi)	Slope (Young's Modulus versus Volume Fraction Porosity)	Extrapolated 100% Density Shear Modulus (10^6 psi)	Slope (Shear Modulus versus Volume Fraction Porosity)
POCO 5Q	2.739	1.660	1.026	1.488
POCO 9Q	6.593	4.040	2.150	3.699
ATJS	2.627	1.804	1.061	1.720

TABLE 5. TEMPERATURE OF INTERNAL FRICTION MINIMUM

Graphite Grade	Approximate Temperature of Internal Friction Minimum (°C)
POCO 5Q	800
POCO 9Q	1000
ATJS	1400

It should also be noted that graphitization of the POCO graphite at 2900°C (POCO 9Q) instead of 2500°C (POCO 5Q) apparently resulted in some densification (~ 1%), but a decrease of approximately 12% in the Young's and shear moduli.

The room temperature Young's moduli of the ATJS specimens (1.604 to 1.805 X 10^6 psi) were near the reported value[3] (1.73 X 10^6 psi) for the room temperature modulus for that material with the grain. The measured room temperature moduli of the POCO 5Q specimens (1.811 to 1.880 X 10^6 psi) were considerably above the reported[4] value (1.68 X 10^6 psi). No data were found for comparison with the results obtained on he POCO 9Q specimens.

The small spread of the elastic moduli values obtained on a given specimen from determination of resonant frequencies with vibration in various directions was taken as evidence of a low degree of anisotropy in all of the materials tested (see Table 3).

ELEVATED TEMPERATURE ELASTICITY AND INTERNAL FRICTION

As shown in the summary Figures 18 and 19, the Young's and shear moduli of all of the specimens increased with increasing temperature, however, the amount of increase and the temperature at which a maximum was observed depended on the graphite grade and on the individual sample. The order of increasing temperature dependence of the moduli and temperature of the moduli maxima was POCO 5Q, POCO 9Q, ATJS.

A hysteresis was observed in the moduli on cooling, as shown in Figures 4–10. Runs to lower maximum temperatures indicated that the hysteresis exists on heating to even a few hundred degrees centigrade, *e.g.,* 300°C, and that the magnitude of the hysteresis is dependent on the maximum temperature achieved. No change was noted in the amount of hysteresis with time at a given temperature up to a few hours. This behavior is consistent with the behavior of other materials with an extreme anisotropy in thermal expansion,[5] and is believed to be due to healing of microcracks on heating, which require some degree of "undercooling" to reopen or reinitiate the same amount of cracks to achieve the same lowering of the elastic modulus on cooling.

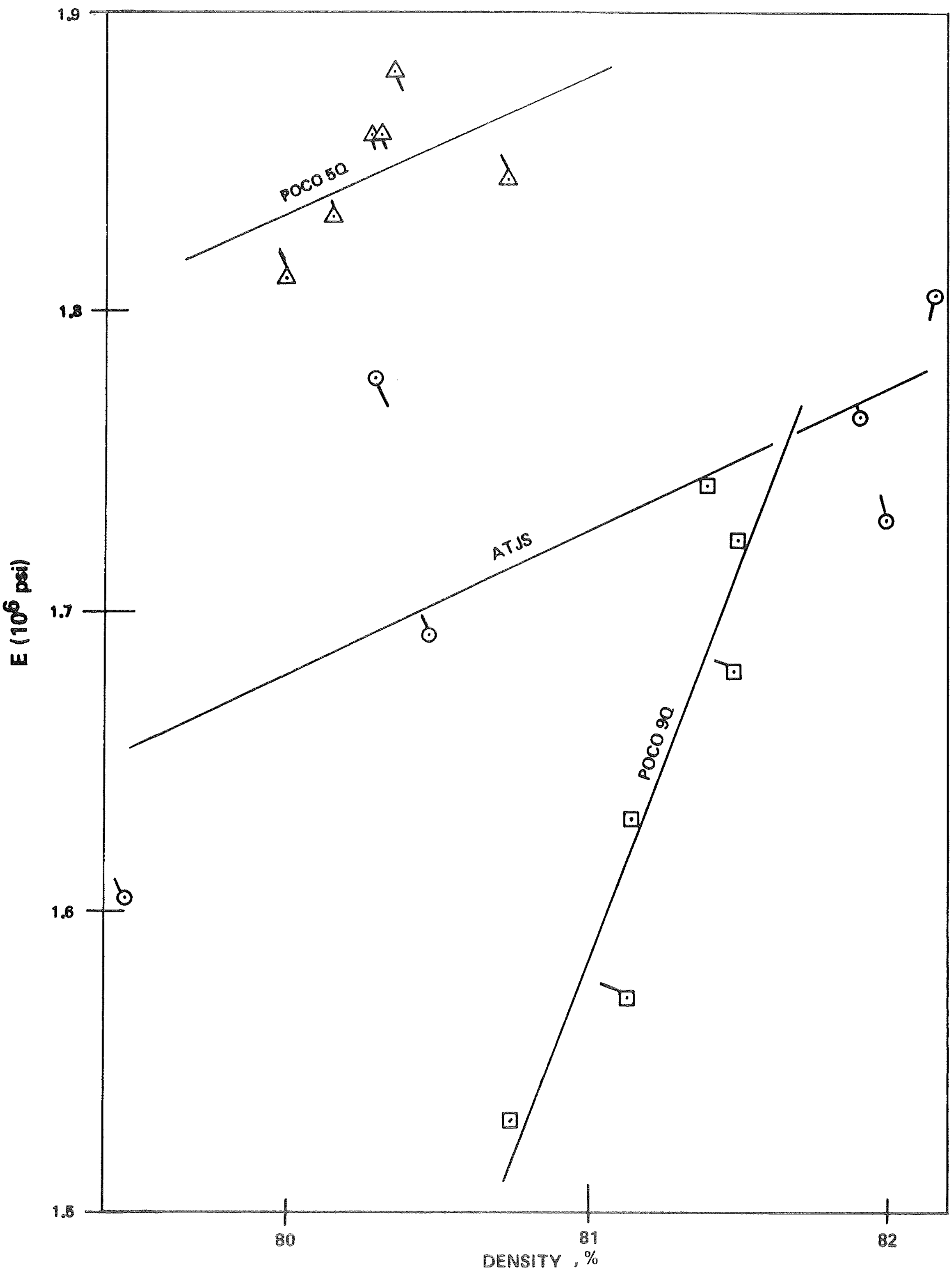

FIGURE 1. DENSITY DEPENDENCE OF ROOM TEMPERATURE YOUNG'S MODULUS. (Lines are linear least–squares fits to data.)

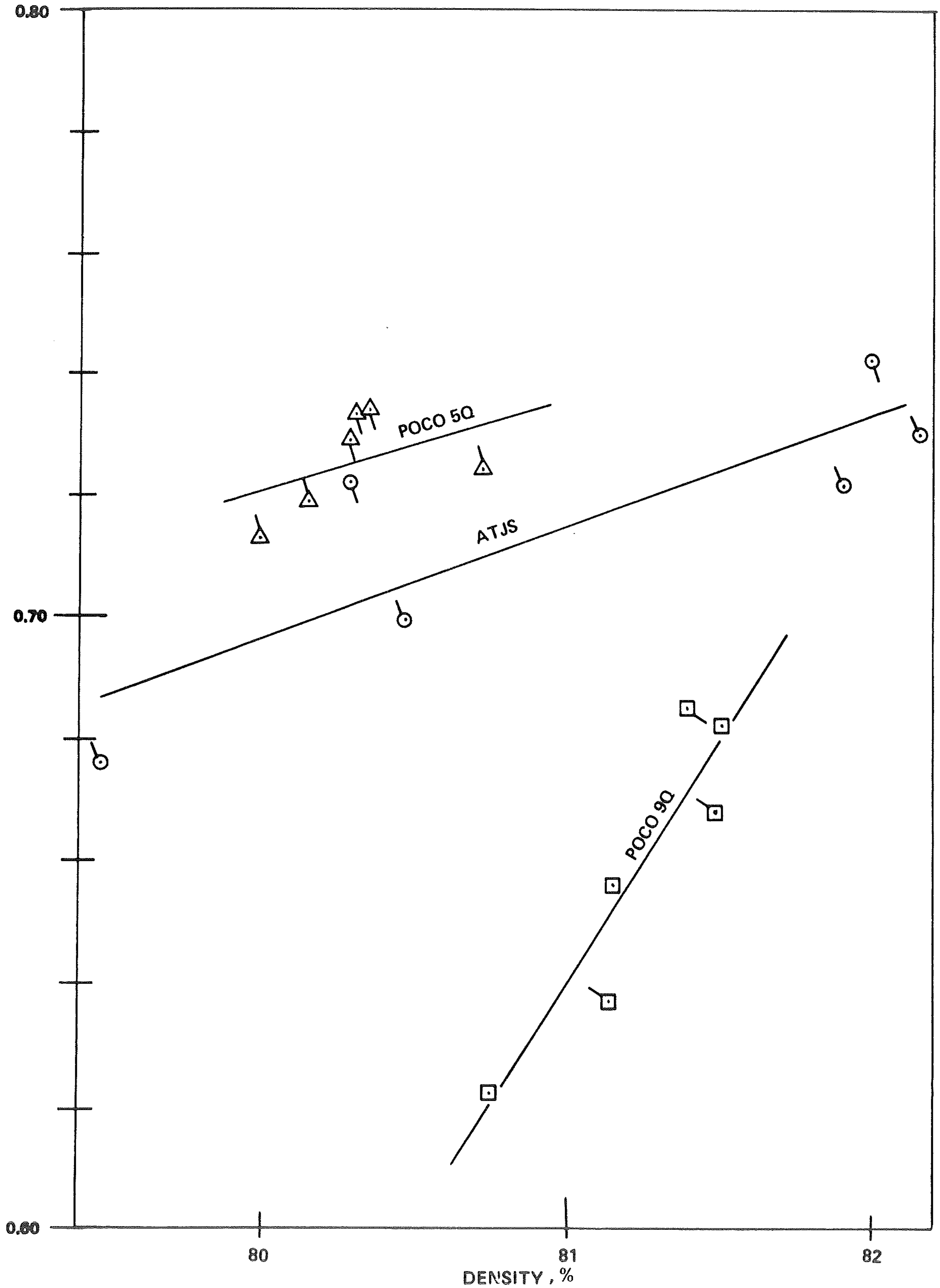

FIGURE 2. DENSITY DEPENDENCE OF ROOM TEMPERATURE SHEAR MODULUS. (Lines are linear least–squares fits of data.)

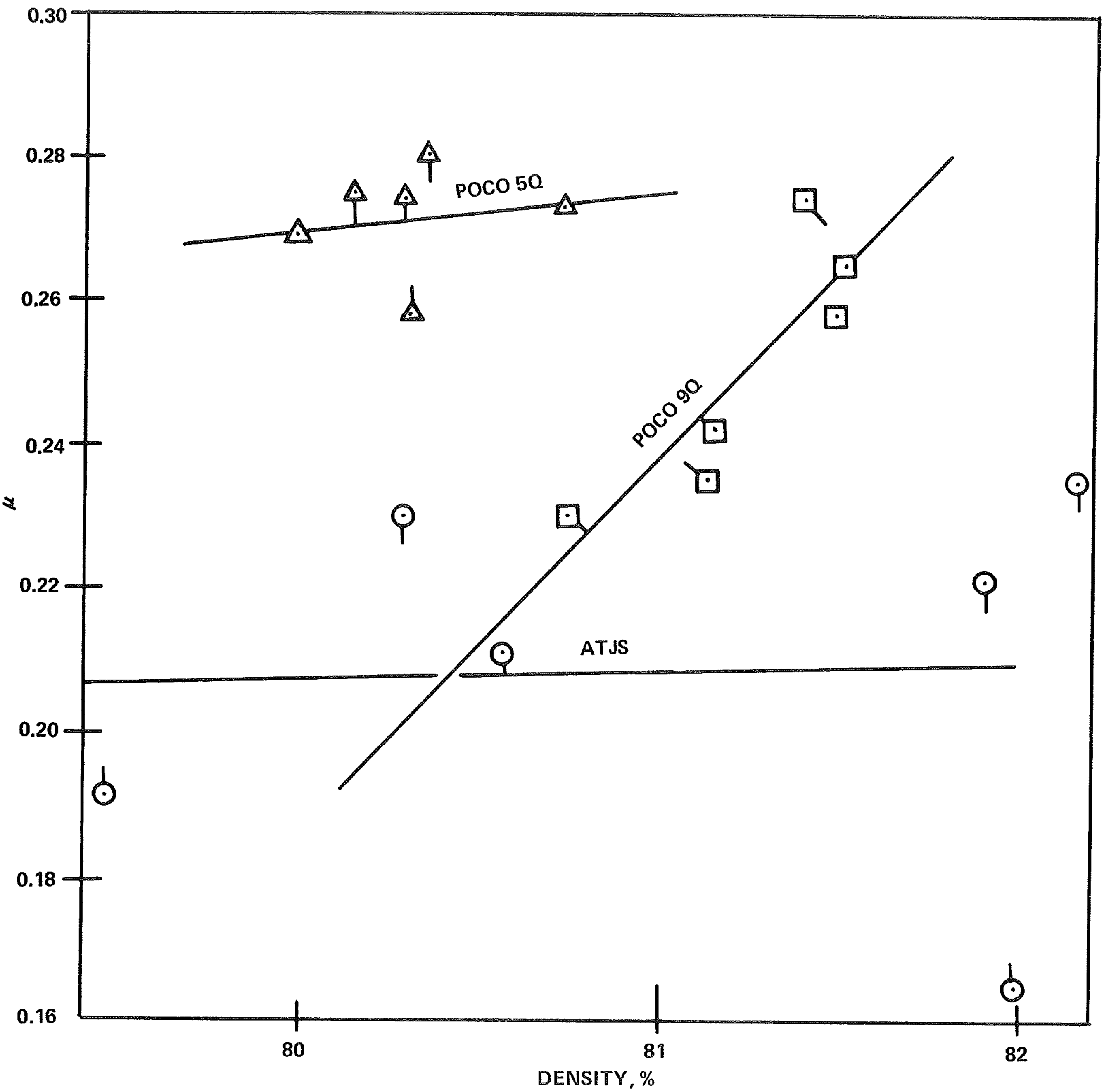

FIGURE 3. DENSITY DEPENDENCE OF ROOM TEMPERATURE POISSON'S RATIO. (Lines are linear least–squares fits to data.)

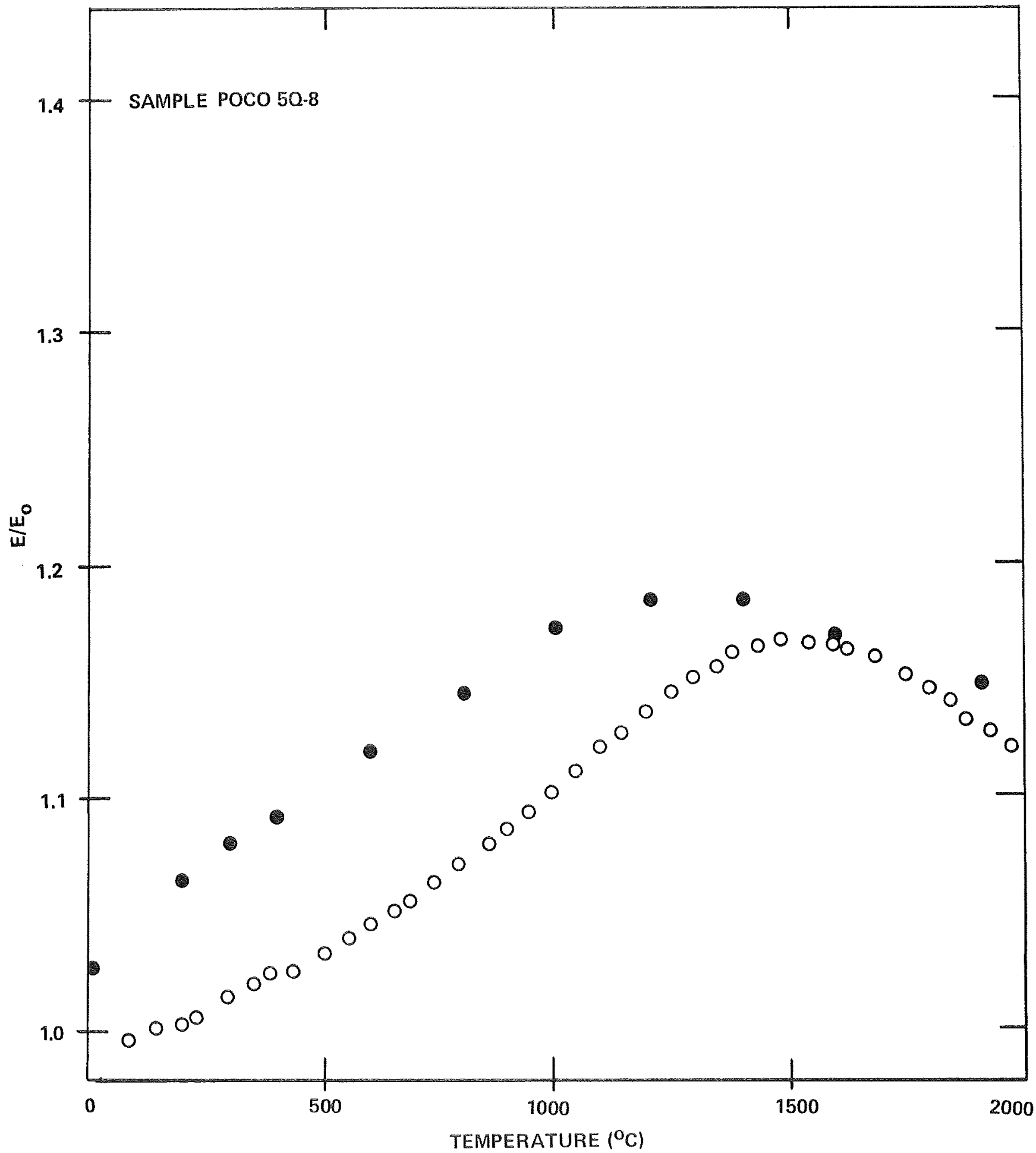

FIGURE 4. TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF SPECIMEN POCO 5Q–8. (Open circles are heating data, solid circles are cooling data.)

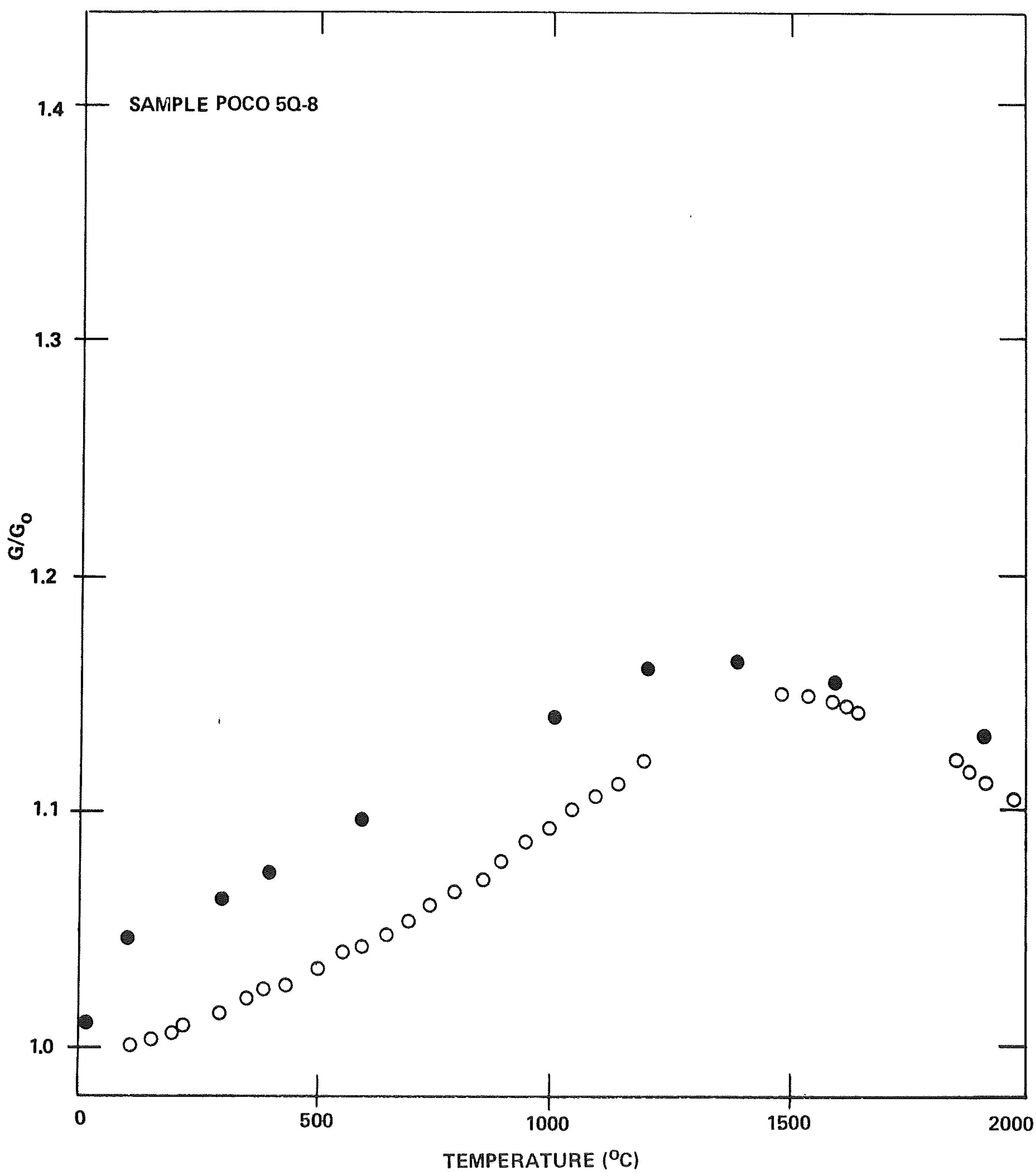

FIGURE 5. TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF SPECIMEN POCO 5Q–8. (Open circles are heating data, solid circles are cooling data.)

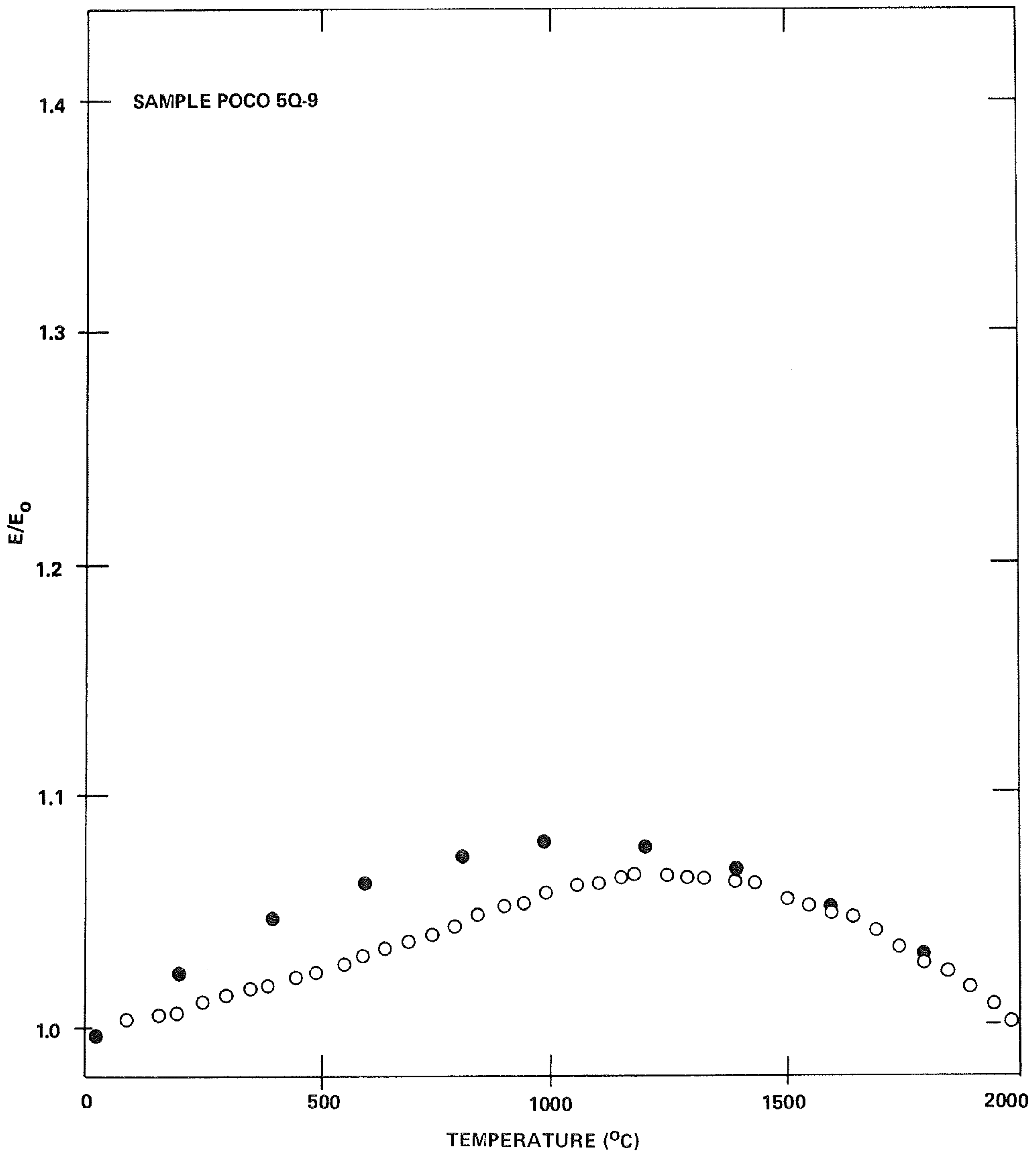

FIGURE 6. TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF SPECIMEN POCO 5Q–9. (Open circles are heating data, solid circles are cooling data.)

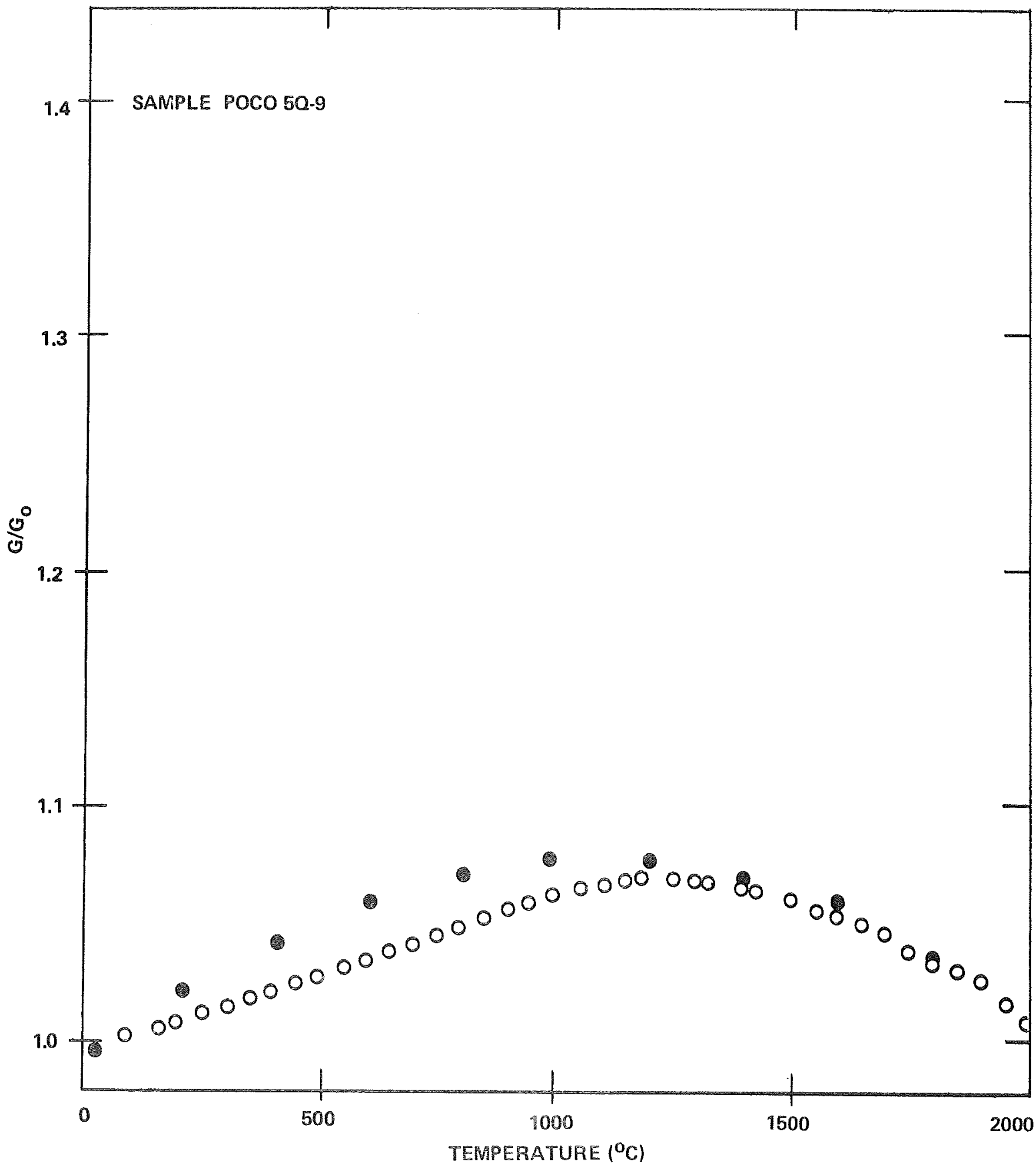

FIGURE 7. TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF SPECIMEN POCO 5Q–9. (Open circles are heating data, solid circles are cooling data.)

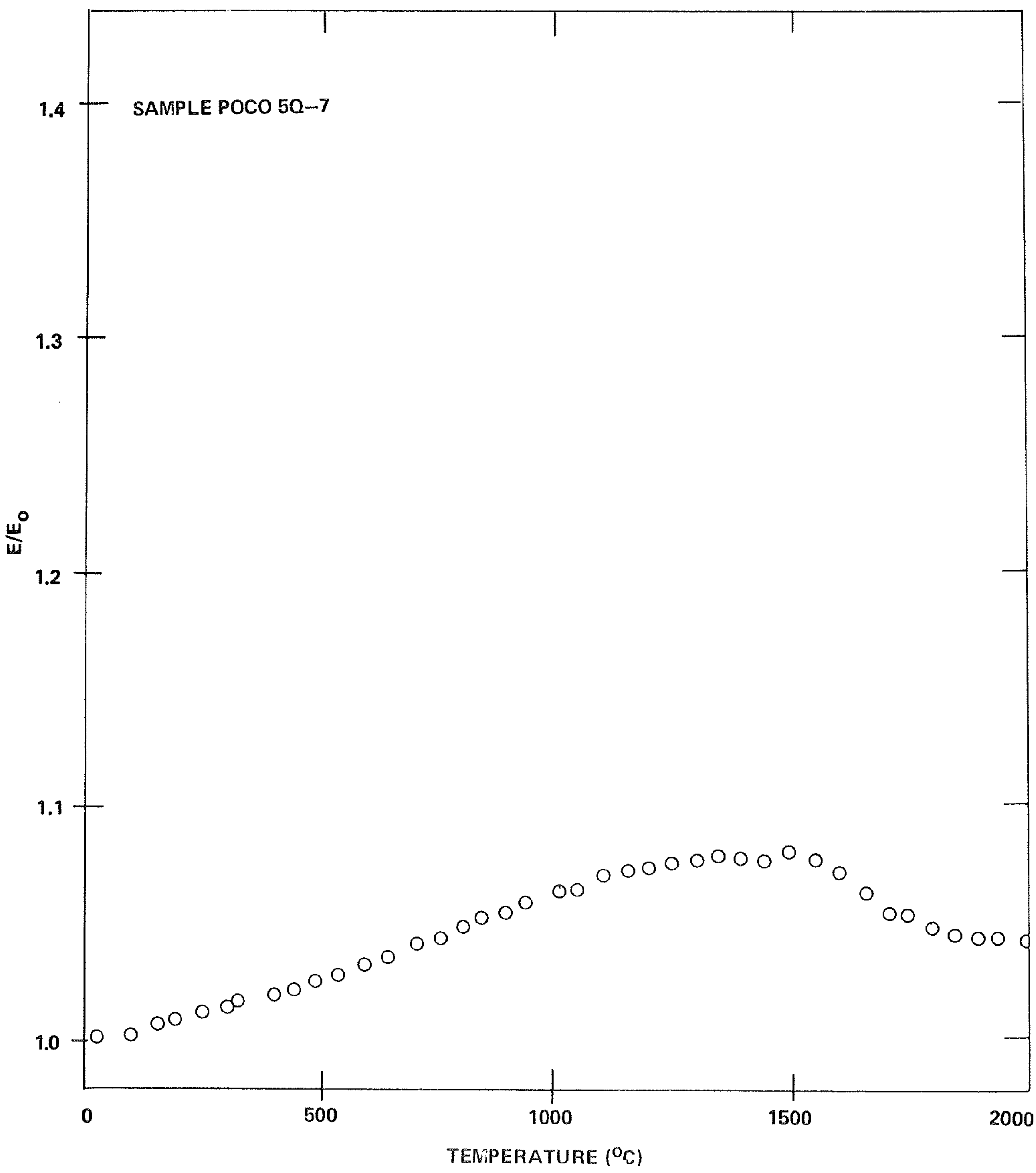

FIGURE 8. TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF SPECIMEN POCO 5Q–7. (Heating data only.)

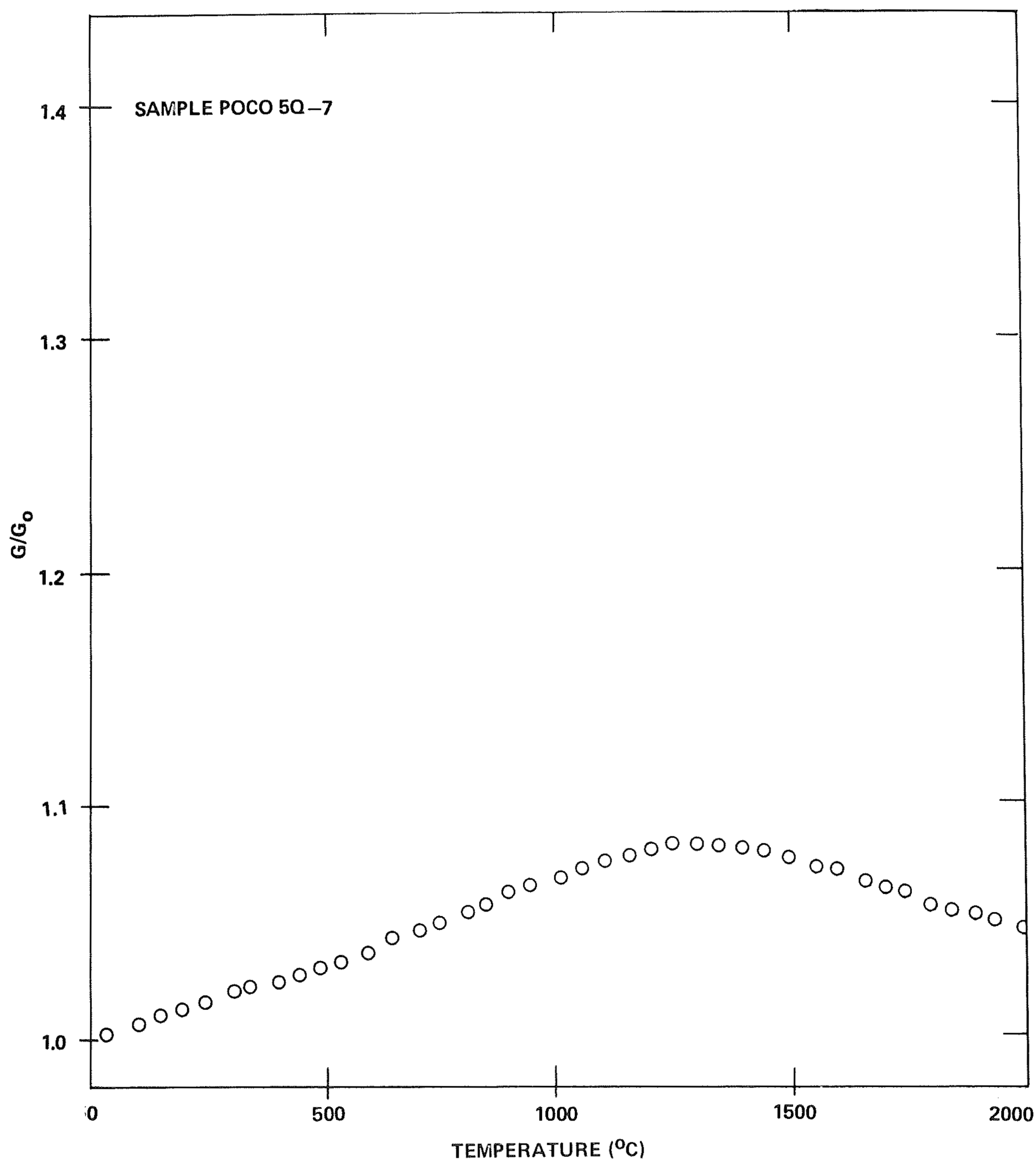

FIGURE 9. TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF SPECIMEN POCO 5Q–7. (Heating data only.)

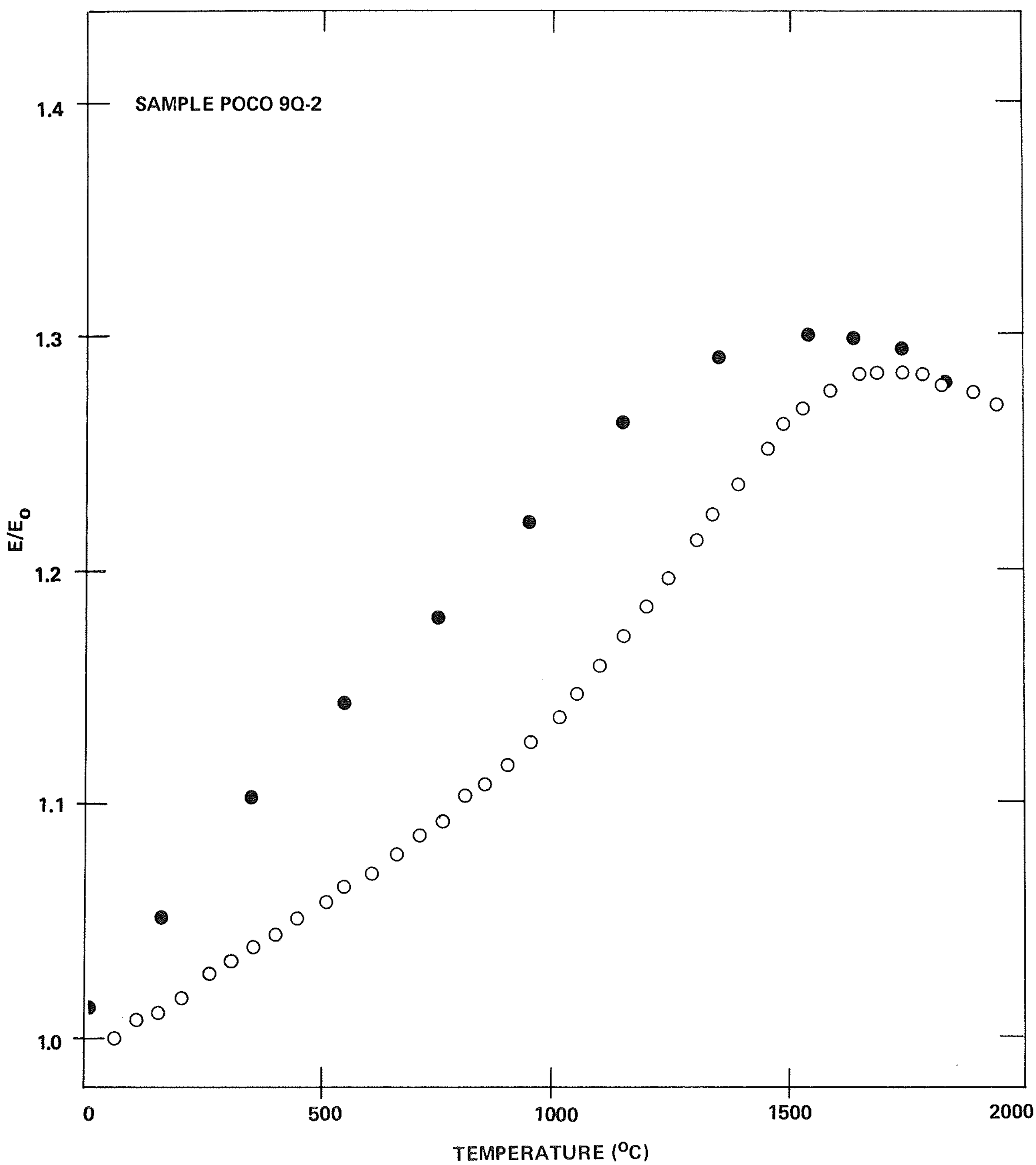

FIGURE 10. TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF SPECIMEN POCO 9Q–2. (Open circles are heating data, solid circles are cooling data.)

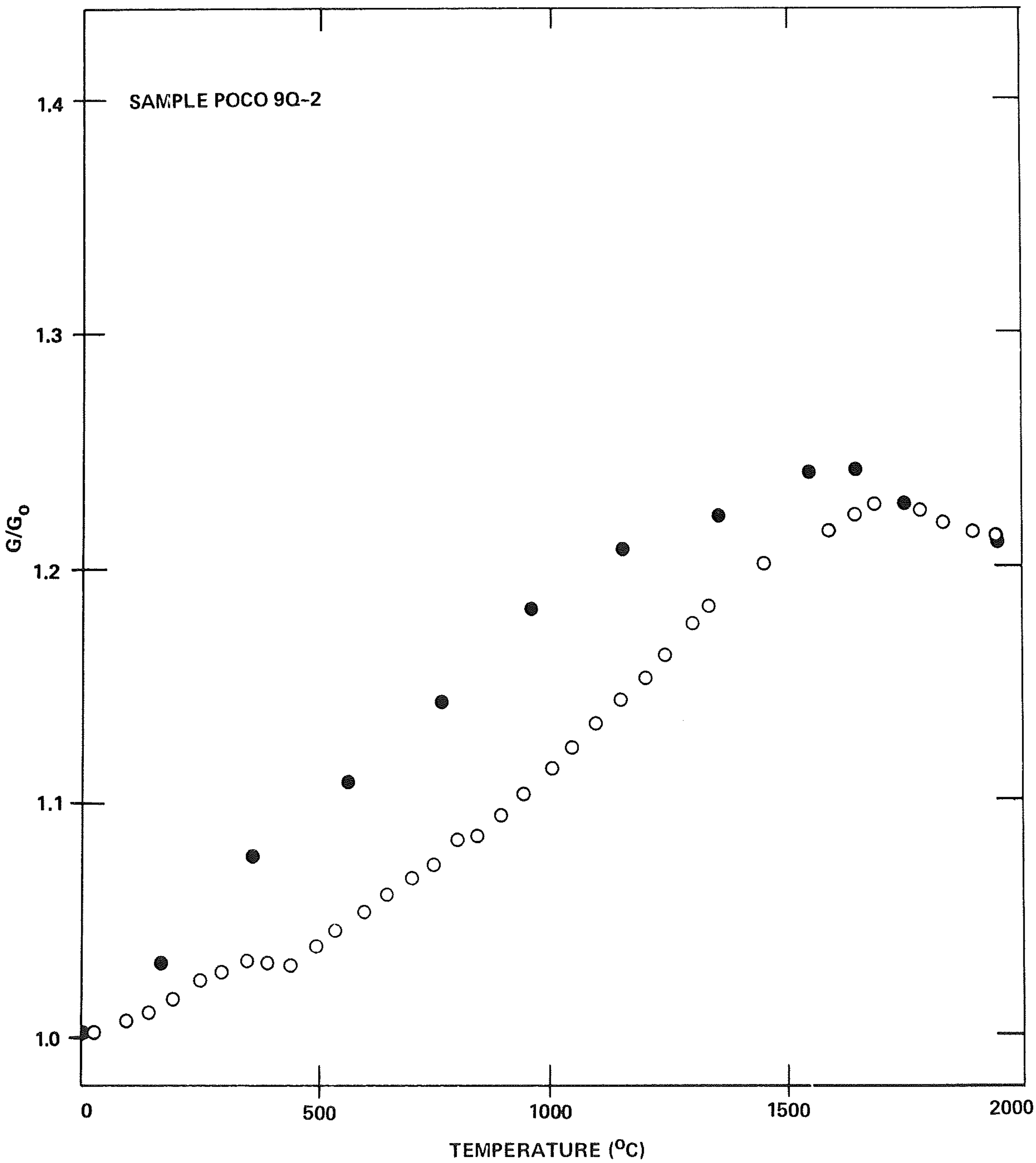

FIGURE 11. TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF SPECIMEN POCO 9Q–2. (Open circles are heating data, solid circles are cooling data.)

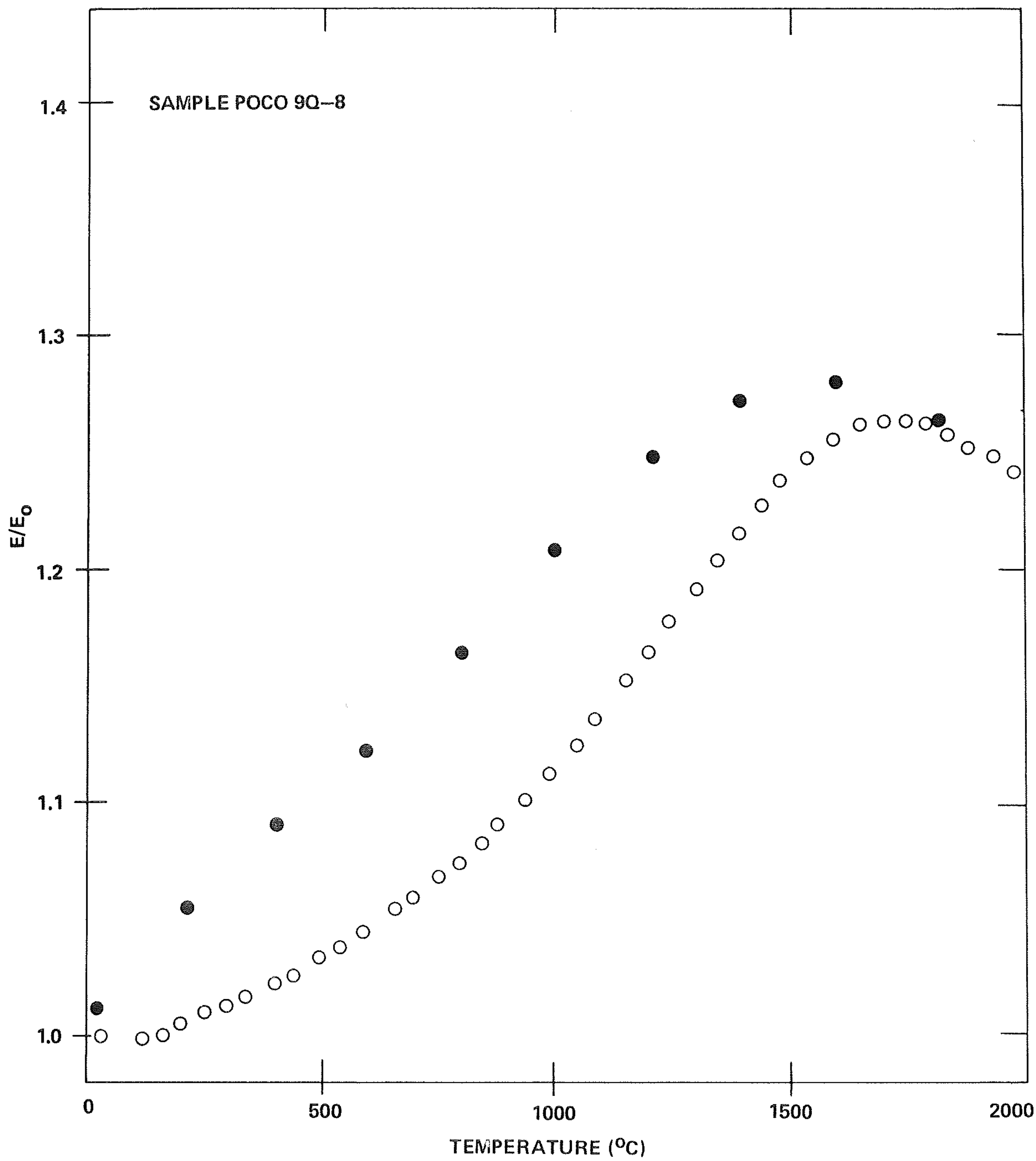

FIGURE 12. TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF SPECIMEN POCO 9Q–8. (Open circles are heating data, solid circles are cooling data.)

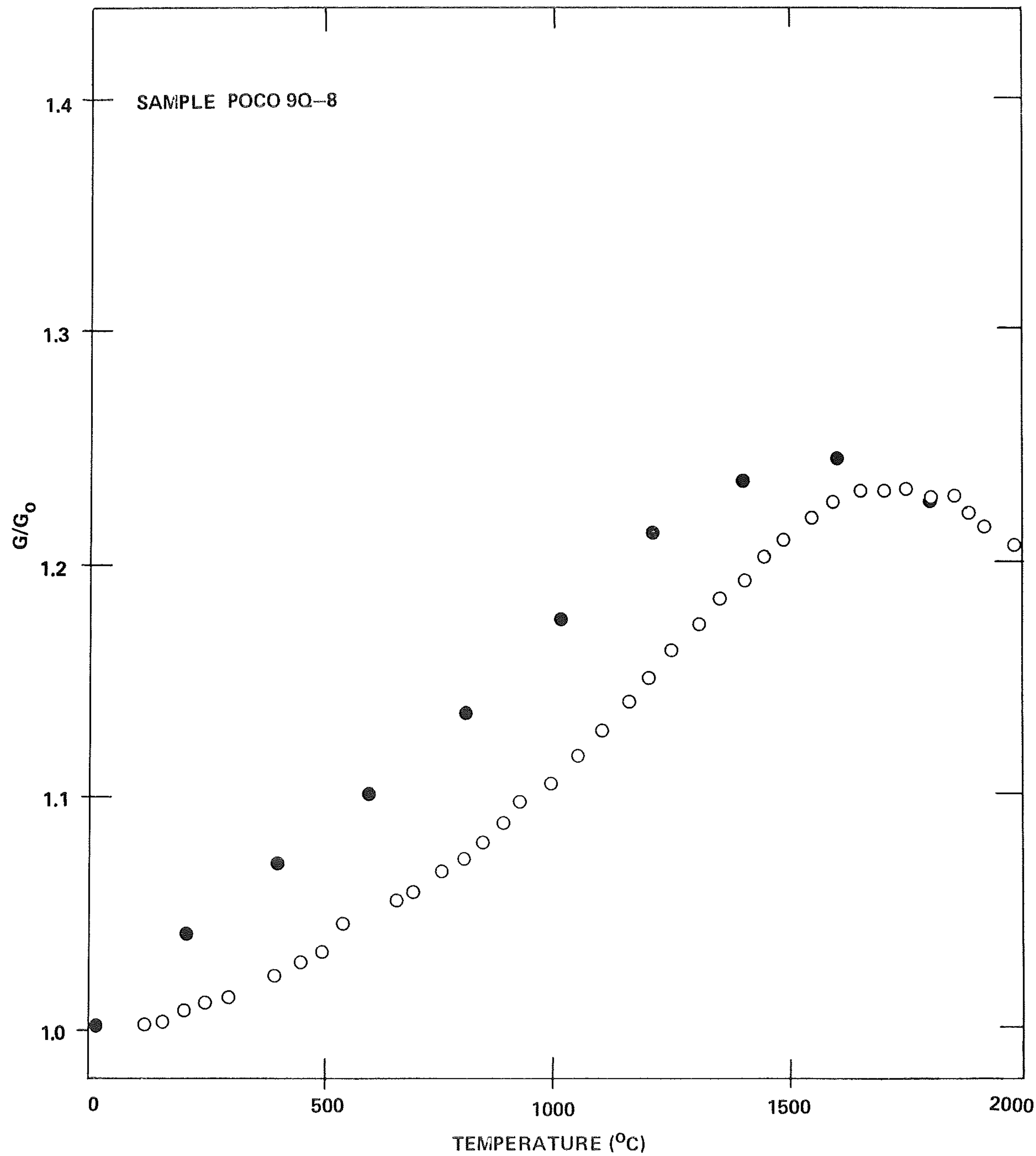

FIGURE 13. TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF SPECIMEN POCO 9Q–8. (Open circles are heating data, solid circles are cooling data.)

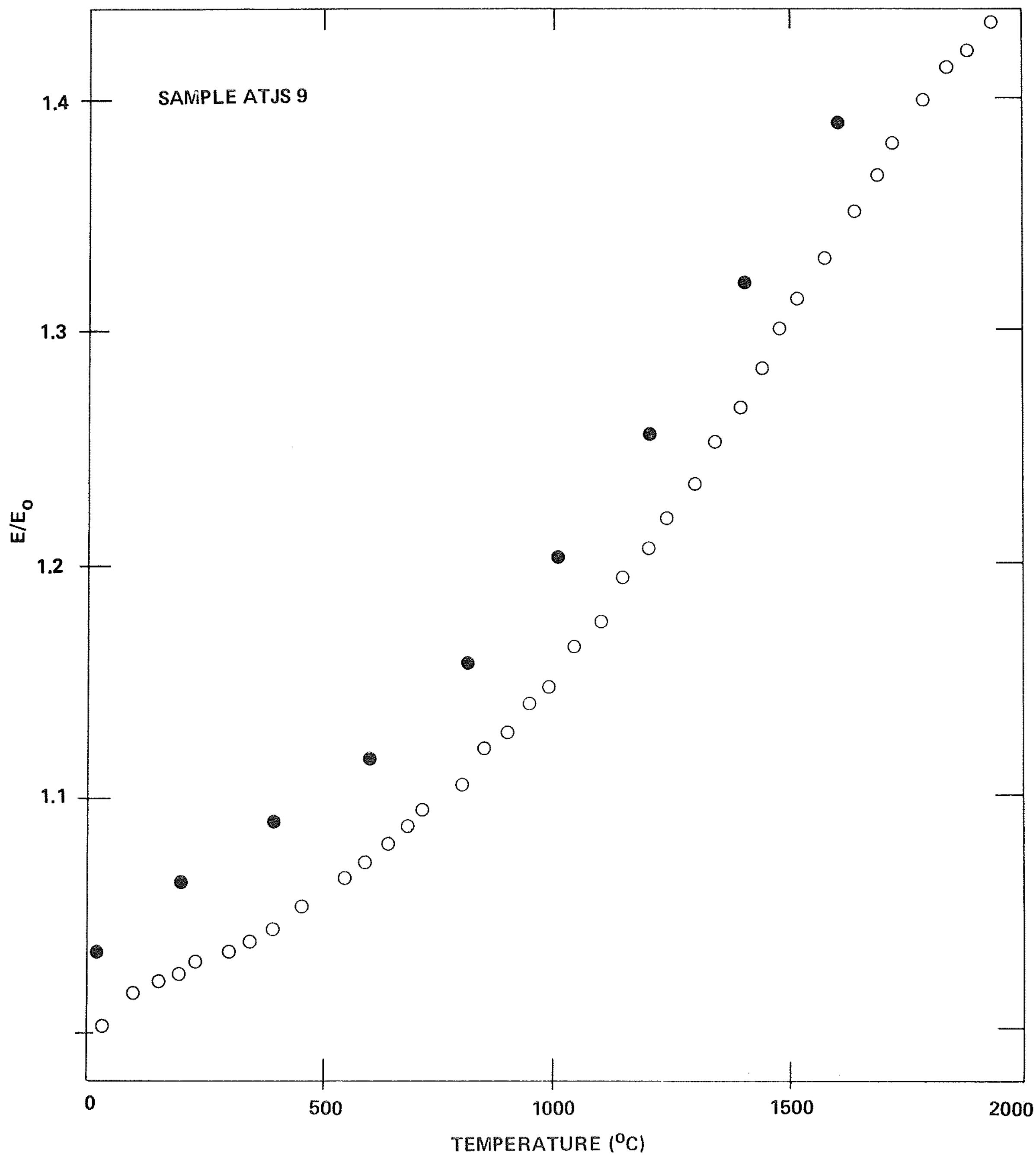

FIGURE 14. TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF SPECIMEN ATJS–9. (Open circles are heating data, solid circles are cooling data.)

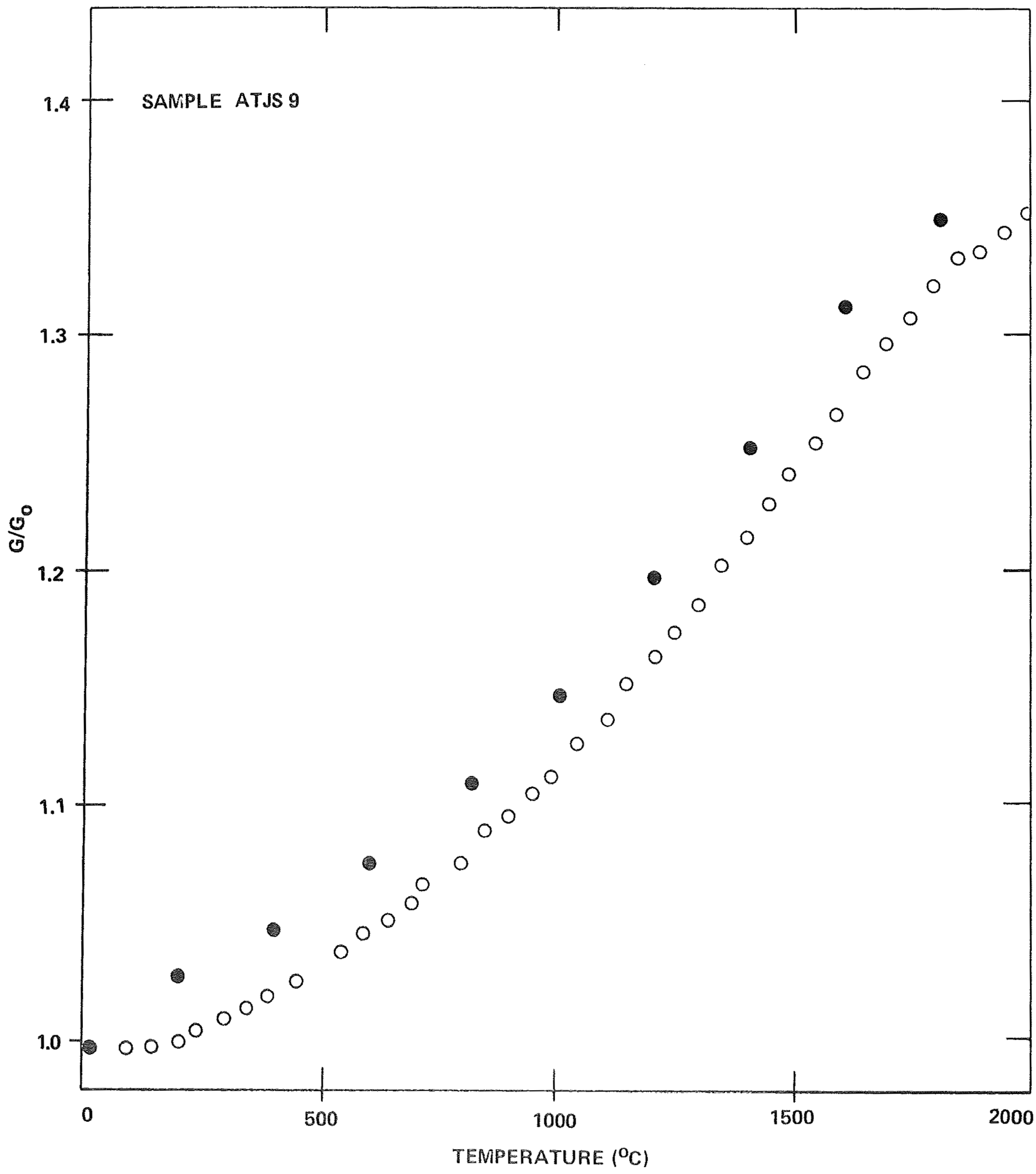

FIGURE 15. TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF SPECIMEN ATJS–9. (Open circles are heating data, solid circles are cooling data.)

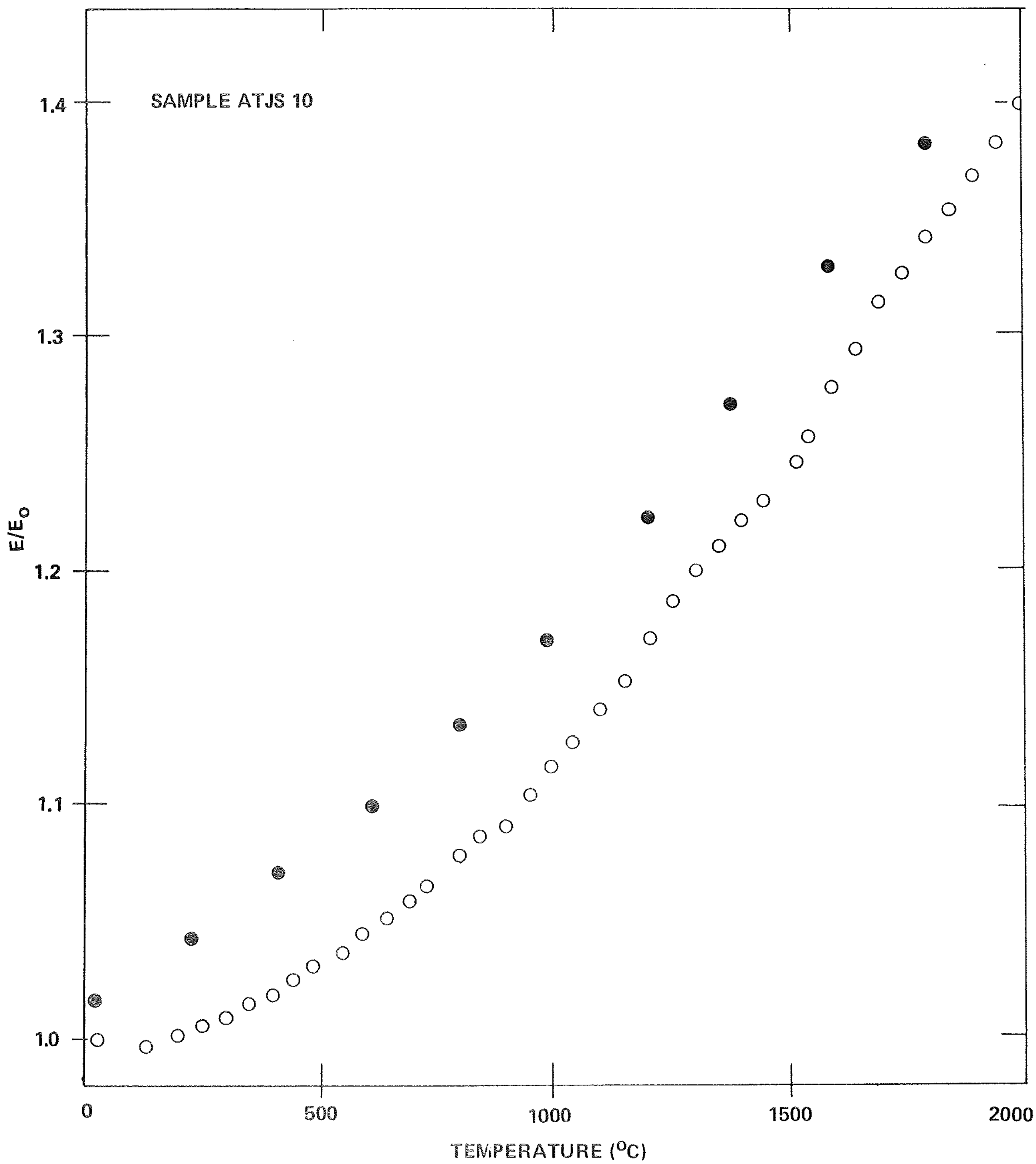

FIGURE 16. TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF SPECIMEN ATJS–10. (Open circles are heating data, solid circles are cooling data.)

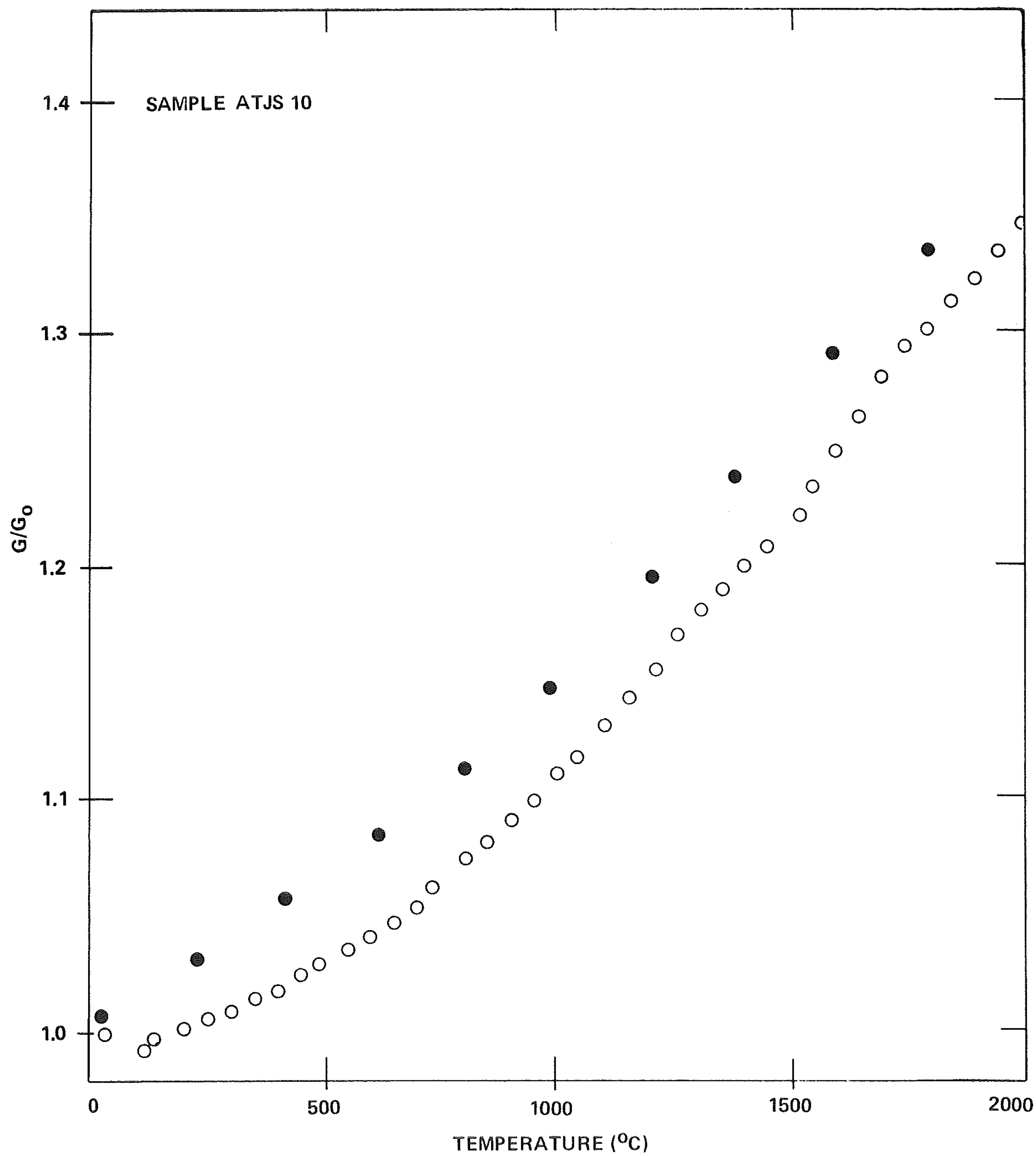

FIGURE 17. TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF SPECIMEN ATJS—10. (Open circles are heating data, solid circles are cooling data.)

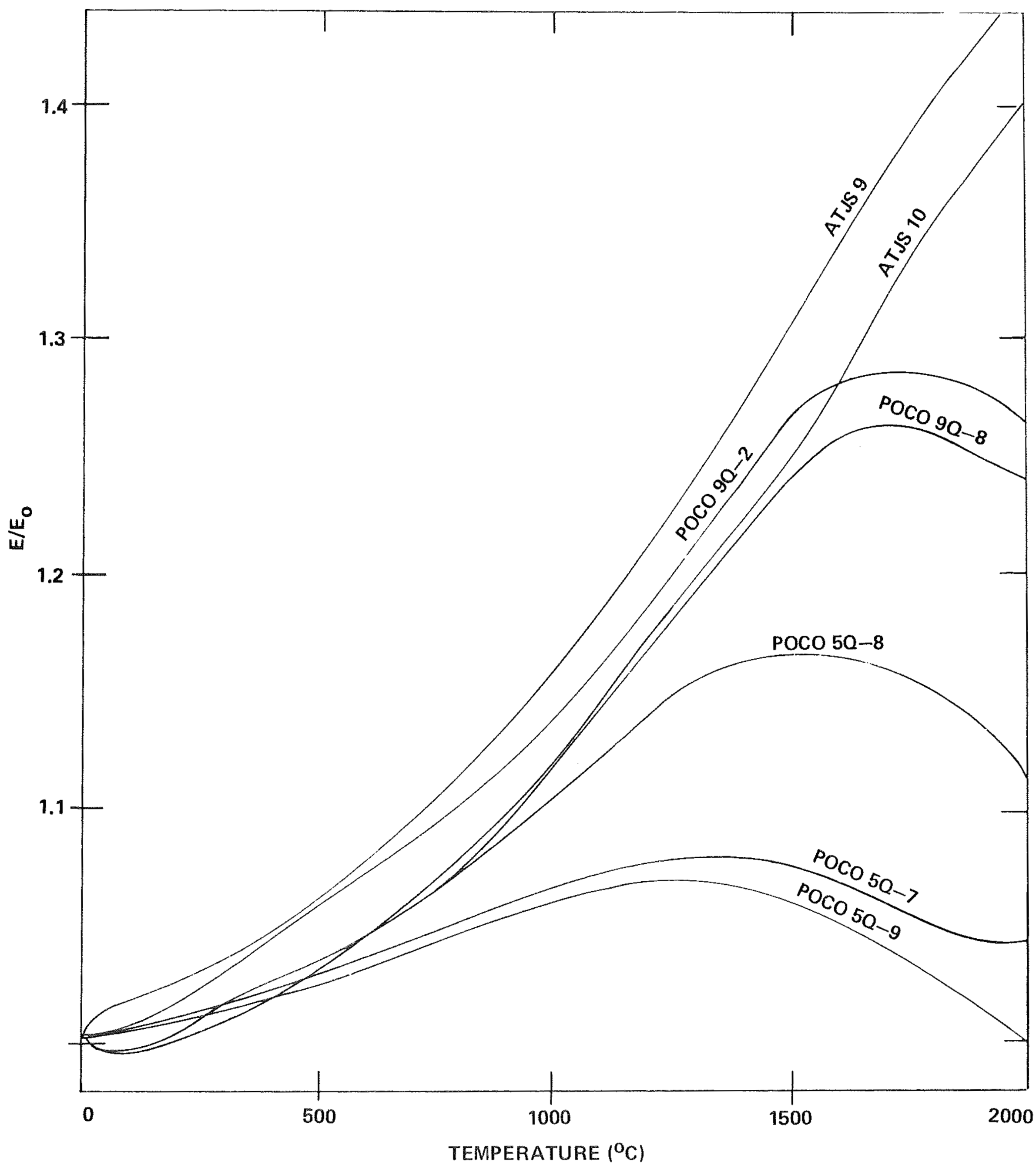

FIGURE 18. SUMMARY OF TEMPERATURE DEPENDENCE OF YOUNG'S MODULUS OF THE THREE GRAPHITE GRADES. (Heating data only.)

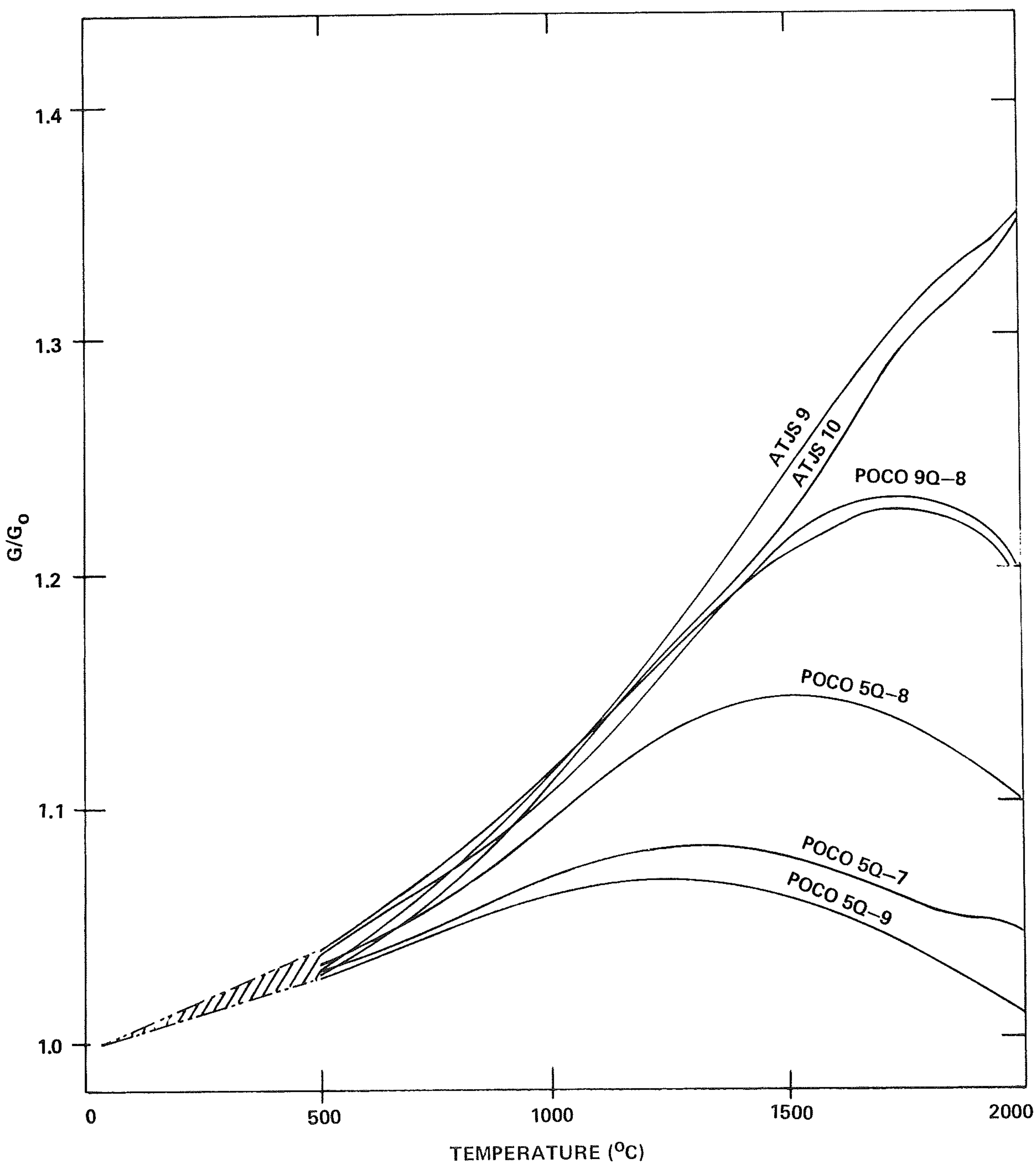

FIGURE 19. SUMMARY OF TEMPERATURE DEPENDENCE OF SHEAR MODULUS OF THE THREE GRAPHITE GRADES. (Heating data only.)

The ATJS graphite exhibited the greatest rate of increase of Young's and the shear modulus with temperature and to 2000°C, no tendency to reach a maximum value was noted. The rate of increase was intermediate to that reported[3] for ATJS material from tensile test data with the grain and across the grain. The variation in the increase in moduli between the two samples tested (ATJS–9 and ATJS–20) was ~ 13% at 2000°C.

The moduli of the POCO 9Q graphite increased nearly as rapidly with temperature as the ATJS, however, maximum values were achieved at ~ 1725°C. A considerable difference was again noted between samples, amounting to ~ 10% at 2000°C.

The moduli of the POCO 5Q graphite exhibited the slowest rate of increase with temperature and the lowest temperature of the maximum moduli values, varying from 1250 to 1500°C for the three samples tested. The variation of the temperature dependence of these elastic moduli from sample to sample was remarkable, however. (See sample POCO 5Q–8, POCO 5Q–9 and POCO 5Q–7, Figures 11 and 12).

CALCULATION OF STATIC MODULI

At the initiation of this study it was proposed that the static (isothermal) elastic moduli of the graphite materials tested be calculated from the dynamic results.[6] However, the uncertainty in the porosity dependence of the moduli, the effect of microcracks on the moduli,[7] the sample to sample variation of the temperature dependence of the moduli, the hysteresis in the moduli on heating and cooling, uncertainties in thermal expansion of the various graphite grades, and lack of accurate heat capacity data on the graphites makes this calculation impractical.

CONCLUSIONS

The results of this study indicate that:

1. Over at least a limited range, the Young's modulus and the shear modulus of all three grades of graphite (POCO 5Q, POCO 9Q and ATJS) are directly proportional to density.

2. On heating, the moduli of all three grades increase with temperature, the rate of increase increasing in the order POCO 5Q, POCO 9Q and ATJS.

3. On continued heating a maximum value of the Young's modulus and shear modulus are achieved, after which the moduli decrease with continued heating. The temperature of the maxima are for POCO 5Q, 1250 to 1550°C; for POCO 9Q, 1700 to 1750°C; and for ATJS, no maximum was observed below 2000°C.

4. There is considerable variation in the temperature dependence of the moduli from sample to sample of the same grade.

5. A hysteresis is observed in the Young's and shear moduli of all three grades of graphite on heating and cooling. The hysteresis exists on heating to only a few hundred degrees centigrade and does not change in magnitude on continued heating up to a few hours.

RECOMMENDATIONS FOR FURTHER STUDY

The results and conclusions of this study indicate several possible areas where refinements of measurements would be fruitful. These should include:

1. Improved definition of the density (porosity) dependence of elasticity through measurement of statistically significant samples covering a wide range of densities.

2. Determination of the effect of heat treatment on the hysteresis of the elastic moduli on heating and cooling.

3. Extension of the measurements to higher temperatures.

4. Statistical analysis of the variation of temperature dependence of elasticity for each graphite grade and determination of controlling processing parameters and possible heat treatments to effect a uniform behavior.

5. Accurate measurement and comparison of the thermal expansion behavior of the graphite specimens.

ACKNOWLEDGMENTS

The author expresses his gratitude to K. H. Peterson who made most of the measurements, J. O. Hegland who helped in setting up the equipment, and H. C. Lockhart who drew the figures.

REFERENCES

1. F. Forster, "New Method for the Determination of the Modulus of Elasticity and Damping." *Z. Metallkunde,* **29**, 109–15, (1937).

2. S. Spinner and W. E. Tefft, "Method for Determining Mechanical Resonance Frequencies and for Calculating Elastic Moduli from these Frequencies," *Proc. ASTM,* **61**, 1221–38, (1961).

3. Technical Information Bulletin No. 463–201 HI, Union Carbide Corp., Carbon Products Division, 270 Park Ave., New York, N. Y. 10017.

4. Technical Data Sheet PGI–8s–674, POCO Graphite, Inc., Garland, Tex. 75041.

5. E. A. Bush and F. A. Hummel, "High–Temperature Mechanical Properties of Ceramic Materials; II. Beta–Eucryptite." *J. Am. Ceram. Soc.,* **42**, 388–391, (1959).

6. M. O. Marlowe, "High Temperature Isothermal Elastic Moduli of UO_2, *J. Nucl. Matl.,* **33**, 242–244, (1969).

7. D. P. H. Hasselman, "Voigt and Reuss Moduli of Polycrystalline Solids with Microcracks," *J. Am. Ceram. Soc.,* **53**, 170, (1970).

DISTRIBUTION LIST

	Copies
NASA Langley Research Center Hampton, Virginia 23365	
Attention: Research Program Records Unit,, Mail Stop 122	1
Raymond L. Zavasky, Mail Stop 117	1
James P. Howell, Mail Stop 335	25
Veryl D. Crowder, Mail Stop 358	1
Howard G. Maahs, Mail Stop 214	1
NASA Ames Research Center Moffett Field, California 94035 Attention: Library, Mail Stop 202-3	1
NASA Flight Research Center P.O. Box 273 Edwards, California 93523 Attention: Library	1
Jet Propulsion Laboratory 4800 Oak Grove Drive Pasadena, California 91103 Attention: Library, Mail 111-113	1
NASA Manned Spacecraft Center 2101 Webster Seabrook Road Houston, Texas 77058 Attention: Library, Code BM6	1
NASA Marshall Space Flight Center Huntsville, Alabama 35812 Attention: Library	1
NASA Wallops Station Wallops Island, Virginia 23337 Attention: Library	1
NASA Lewis Research Center 21000 Brookpark Road Cleveland, Ohio 44135 Attention: Library, Mail Stop 60-3	1
NASA Goddard Space Flight Center Greenbelt, Maryland 20771 Attention: Library	1
NASA John F. Kennedy Space Center Kennedy Space Center, Florida 32899 Attention: Library, Code IS-CAS-42B	1

National Aeronautics and Space Administration Washington, D.C. 20546 Attention: Library, Code USS-10 NASA Code S	 1 1
POCO Graphite, Inc. Director of Research Box 2121, Decatur, Decatur, Texas 76234 Attention: Dr. R. F. Wehrmann	 1 1
Los Alamos Scientific Laboratory University of California Los Alamos, New Mexico 87544 Attention: Morton C. Smith	 1
McDonnell Douglas Corporation 3000 Ocean Park Boulevard Santa Monica, California 90406 Attention: Dr. Hank W. Babel	 1
Technical Consultant 42 Lookout Drive Dayton, Ohio 45409 Attention: S. W. Bradstreet	 1
Union Carbide Corporation Carbon Products Division 270 Park Avenue New York, New York 10017	 1
General Electric Company Reentry Systems Department Materials Performance Laboratory P.O. Box 8555 Philadelphia, Pennsylvania 19101 Attention: K. J. Hall	 1
IITR Research Institute Applied Ceramics 10 West 35th Street Chicago, Illinois 60616 Attention: S. A. Bortz, Manager	 1
Lockheed Palo Alto Research Laboratory Lockheed Missiles and Space Company Carbon Research and Development Department 3251 Hanover Street Palo Alto, California 94303	 1
NASA Scientific and Technical Information Facility P.O. Box 33 College Park, Maryland 20740	 11 plus reproducible