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MECHANICAL PROPERTIES OF GRAPHITE/EPOXY COMPOSITES AT VARIOUS TEMPERATURES

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Purdue University

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Off-axis tension tests were performed for coupon specimens of unidirectional graphite/epoxy composites at various temperatures. Elastic moduli and principal strengths of the composite were obtained by a simple procedure. To avoid the complication caused by the extension-shear coupling effect in off-axis testing, a new tab was designed and used on the test specimens. Excellent results for tensile modulus, tensile strength, shear modulus, shear strength, and Poisson's ratio were obtained.

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PREFACE

The High Temperature Materials - Mechanical, Electronic and Thermophysical Properties Information Analysis Center (HTMIAC) is a U.S. Department of Defense (DoD) Information Analysis Center sponsored by the Office of the Undersecretary of Defense for Acquisition (Research and Advanced Technology). It is operated by the Center for Information and Numerical Data Analysis and Synthesis (CINDAS), Purdue University, West Lafayette, Indiana 47906, under the Defense Logistics Agency (DLA) Contract DLA900-86-C-0751. The DLA contract is awarded to Purdue by the Defense Electronics Supply Center (DESC), Dayton, Ohio 45444-5208, with Ms. Sara M. Williams and Mrs. Karen M. Colwell as the Contracting Officers.

HTMIAC is under the technical direction of Mr. Jerome Persh, Office of the Undersecretary of Defense for Acquisition (Research and Advanced Technology), Attn: OUSDA (R&AT)(ET), Pentagon, Room 3D1089, Washington, D.C. 20301-3081 and Dr. Jim C. I. Chang, Naval Research Laboratory, Code 6330, Washington, D.C. 20375-5000. HTMIAC is under the program management of Mr. Paul M. Klinefelter and Mr. Brian P. McCabe, the Defense Technical Information Center (DTIC), Attn: DTIC-DF, Cameron Station, Alexandria, Virginia 22304-6145.

HTMIAC serves as the DoD's central source of data and information on high temperature materials properties, especially the properties of aerospace structural composites and metals and of infrared detector/sensor materials. It supports the DoD research, development, engineering, and studies programs and weapons systems in general, and supports the DoD Tri-Service Laser Hardened Materials and Structures Group (LHMSG) to meet the material property data requirements for high energy laser structural and detector vulnerability, survivability, and hardening assessments and studies in particular. It also provides similar support to the DoD high energy laser community associated with the Strategic Defense Initiative (SDI) programs.

To fulfill its assigned mission to support the DoD, HTMIAC performs both its basic operation and special studies/tasks. The work detailed in this technical report constitutes a part of a special study entitled "Thermophysical and Mechanical Properties of Composite Materials under Rapid Heating Conditions" sponsored by the Defense Nuclear Agency (DNA), Washington, D.C. 20305-1000, through DNA MIPR 87-617. The DNA Program Managers are Major Gilbert Wendt, Attn: DNA-SPAS, and Major Ken Zeringue, Attn: DNA-SPSD.

This part of the Special Study was performed by the Composite Materials Laboratory (CML) of the School of Aeronautics and Astronautics of Purdue University under the supervision and technical administrative support of HTMIAC. Well known in this country and abroad, the Composite Materials Laboratory at Purdue is one of the best research laboratories on composite materials in the United States.

C. Y. Ho
Director
HTMIAC and CINDAS

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1. Introduction

Failure in composite laminates subjected to intense heat has been a subject of interest [1,3]. In failure analysis temperature dependent material properties are required. For epoxy-based composites, the temperature range of practical interest lies between 20°F–500°F. Beyond 500°F, the matrix as well as the composite stiffness and strength diminish. Although fibers retain their tensile properties way above 500°F, their compressive and shear properties become insignificant. From the laminate strength point of view, it is not important to measure composite properties beyond 500°F.

In this report, a simple method for testing composite stiffness and strength properties was proposed and validated. This method is based on testing off-axis coupon specimens with a newly designed flexible tab which allows shear deformation to avoid the extension-shear coupling effect. This method is particularly convenient to use in elevated temperature environments.

2. Experimental Procedures

Specimens and Tabs

Panels of $[0]_{10}$ and $[0]_{16}$ graphite/epoxy were fabricated with Hercules AS4/3501-6 prepreg tapes. The 0° and 15° coupon specimens were cut from the $[0]_{10}$ panel. The 45° and 90° specimens were cut from the $[0]_{16}$ panel. The specimen length was 8.0 inch and the width was 1.0 ± 0.01 inch.

Rigid glass/epoxy tabs were used in testing 0° and 90° coupon specimens where extension shear coupling is absent; both the rigid tabs and the newly designed flexible tabs were used in testing 15° and 45° coupon specimens where extension shear coupling exists. The shear properties using these two kinds of tabs were compared.

New Tab Design for Off–axis Testing

A number of different test methods are currently in use to obtain shear properties of composite materials. Rail shear, torsional tube, $[\pm 45]$ coupon tests, and the off-axis coupon are the typical shear testing methods commonly used. Each test has its own advantages and disadvantages.

Among these methods, the off-axis coupon test seems ideal because of its easy specimen fabrication, very simple data reduction, and no special equipment requirement. In order to extract material constants from an off-axis test, it is assumed that a uniform state of both stress and strain exists throughout the test section. One problem with off-axis tests of unidirectional composites is the extension-shear coupling resulting from the anisotropy of the material, since shear deformation is constrained in the conventional simple tension test. This restriction of shear deformation causes high stress concentrations and nonuniformities throughout the stress field in off-axis testing [4,5].

To solve the problem, the load must be applied in a way which allows shear deformation. A tab between the specimen and grip which allows shear deformation would solve the problem. Tests performed with various soft tab materials exposed another problem. The tab must be very stiff transversely, or the grips tend to crush the unidirectional coupon. An orthogonally woven fiberglas cloth impregnated with siliconerubber exhibits all desired characteristics. One of the best fiberglas cloths was a bi-directional E-glass knit (18.17 oz/sq yd) produced by King Fiberglass Corporation. This knit consists of a layer of unidirectional fiberglas laid transversely over another layer of unidirectional fiberglas knit together with polyester thread. The matrix material was composed of an RTV664 heat cured siliconerubber compound produced by General Electric.

Tab Fabrication Procedure

Twelve inch square panels were fabricated using siliconerubber and fiberglas cloth. A layer of release ply was laid on a tool plate. The fiberglas was then cut and laid over the release ply. Four ounces of siliconerubber were then mixed and spread evenly over the cloth. A second layer of release ply and a caul plate were placed over the panel. The panel was then placed in a vacuum bag and put into an autoclave to cure. The resulting panels had a nominal thickness of 0.040 inch.

Stress Distribution Test

To evaluate the new flexible tab, 10° and 20° off-axis coupons of 8 ply graphite/epoxy were used in measuring stress distribution. For comparison purposes, conventional glass/epoxy tabs were also used. Longitudinal strains at four points were measured by mounting Micro Measurement EA-13-125AC-350 strain gages. Positions of the longitudinal strains are shown in Fig. 1.

Tabs cut 1.0 x 1.5 inches were placed on the test coupon and secured with a thin band of tape. The fiberglas knit tabs must be placed on the specimen with the transverse fiber direction next to the specimen. This placement is required to minimize crushing after securing by the hydraulic grips. The tab composed of siliconerubber and fiberglas was incapable of achieving extremely high loads. During the test it was found that the tab could transmit loads up to 1500 lbs. Higher loads resulted in the fiberglas knit failing and the specimen slipping out of the grips. Each coupon was placed in an MTS 810 material testing system. The specimen was gripped in MTS 647 hydraulic wedge grips under 600 psi.

Figures 2-a,b,c,d show the strain distribution test results for 10° and 20° off-axis coupon specimens using rigid glass/epoxy tabs and the fiberglas-knit/silicon-rubber tabs, respectively. There are large stress concentrations (see gages 1 and 2 in Figs. 2-b and d) near the end tabs of the specimens using glass/epoxy tabs. However, Figs. 3-a and b show that a state of uniform strain was achieved by using flexible tabs.

Therefore, it is evident that more exact shear properties can be obtained by using flexible tabs than by using conventional rigid tabs in off-axis tests.

Test Procedures

Uniaxial tension tests were performed using the closed-loop-servo hydraulic MTS 810 machine at 75°F, 250°F, 350°F, and 500°F. The testing system is shown in Fig. 3.

The specimen was heated between two resistive heating plates.

The specimen was gripped in MTS 647 hydraulic wedge grips. The grip pressure was set at 1200 psi for the rigid tab specimen and at 600 psi for the flexible tab specimen to avoid crushing the specimen by the gripping force. The gripping test showed that the 15° coupon specimens were crushed at 800 psi grip pressure.

All tests were performed at a constant cross-head speed of 0.003 inch/min. Longitudinal and transverse strains were measured by a dual sensor strain transducer (Measurement Technology, Inc.). The measured analog signals were converted into digital signals. The signals were stored and analyzed by a micro-computer data system to plot the stress-strain curves.

High Temperature Testing Equipment

Two heating plates were made (4.0 inches) to permit the specimen to be gripped outside the heating region. The schematic for the structure of the heating plate is shown in Fig. 4. The 12 Ω resistive heating wire was wound around an asbestos core. This heating element was insulated with a flexible asbestos sheet and was assembled with two steel plates at each side. The specimen was heated between the two-faced inner steel plates. Two heating elements were connected in serial to a power supply. The temperature was controlled by using a voltage controller with a reference temperature measured at a center hole of the inner heat plate.

The temperature gradients of the heating plate were within ± 5% over 90% of the heater length, and the maximum heating temperature was about 800°F.

Data Evaluation

From the stress-strain curves of 0° and 90° specimens the longitudinal and the transverse properties can be simply obtained. But, to get the shear modulus and strength from an off-axis specimen, some analytical procedures are needed.

- Shear Modulus

The apparent elastic modulus E_x for an orthotropic material stressed at an angle θ to the principal material direction can be related as

$$\frac{1}{E_x} = \frac{1}{E_1}\cos^4\theta + \left[\frac{1}{G_{12}} - \frac{2\nu_{12}}{E_1}\right]\sin^2\theta\cos^2\theta + \frac{1}{E_2}\sin^4\theta \tag{1}$$

where E_1, E_2 are longitudinal and transverse moduli, respectively, and ν_{12} is the Poisson's ratio. From equation (1) shear modulus G_{12} can be written as

$$G_{12} = \frac{1}{\left\{\dfrac{2\nu_{12}}{E_1} + \dfrac{1}{\sin^2\theta\cos^2\theta}\left[\dfrac{1}{E_x} - \dfrac{\cos^4\theta}{E_1} - \dfrac{\sin^4\theta}{E_2}\right]\right\}} \tag{2}$$

E_1, E_2 and ν_{12} can be obtained from 0° and 90° specimen tests, and E_x can be obtained from the θ degrees off-axis specimen tests. Then, G_{12} can be calculated from equation (2).

- Shear Strength

The Tsai-Hill criterion is given by

$$\left[\frac{\sigma_{11}}{X}\right] - \left[\frac{\sigma_{11}}{X}\right]\left[\frac{\sigma_{22}}{X}\right] + \left[\frac{\sigma_{22}}{Y}\right]^2 + \left[\frac{\tau_{12}}{S}\right]^2 = 1 \tag{3}$$

where X, Y, and S are longitudinal, transverse and shear strength, respectively, and σ_{11}, σ_{22} and τ_{12} are stresses in the principal axis direction. Equation (3) can be rewritten as

$$S = \left[\frac{\tau_{12}^2}{\left\{1 - \left[\dfrac{\sigma_{11}}{X}\right]^2 - \left[\dfrac{\sigma_{22}}{Y}\right]^2 + \left[\dfrac{\sigma_{11}}{X}\right]\left[\dfrac{\sigma_{22}}{X}\right]\right\}}\right]^{\frac{1}{2}} \tag{4}$$

The longitudinal and transverse strength can be obtained from the simple tension test of 0° and 90° specimens. Under simple tension condition of θ degrees off-axis orthotropic material,

$$\sigma_{11} = \sigma_x \cos^2\theta$$

$$\sigma_{22} = \sigma_x \sin^2\theta \tag{5}$$

$$\tau_{12} = -\sigma_x \cos\theta\sin\theta$$

where σ_x is the stress in loading direction. The ultimate values of σ_{11}, σ_{22} and τ_{12} can be calculated from the apparent failure stress σ_x of the off-axis coupon specimen. Therefore, shear strength can be calculated from equation (4).

3. Results and Discussion

The apparent tensile moduli and strengths of 0°, 90°, 15°, and 45° coupon specimens are summarized in Table 1, and the evaluated elastic moduli and the strengths are presented in Table 2 and Table 3, respectively. The temperature effects on tensile moduli and strengths are shown in Figs. 5-a,b,c and Figs. 6-a,b,c, respectively.

Modulus

Figures 5-a, b, and c show that the longitudinal modulus does not change with respect to temperature variation, but the transverse and the shear moduli drop to 72-84% of the modulus of room temperature at 250°F, and to 7-10% at 350°F. It is evident that the longitudinal modulus does not change since it is affected dominantly by graphite fiber which is not sensitive to temperature variation. Since the transverse and shear properties are dominantly affected by epoxy matrix, which is sensitive to temperature variation, the shear and the transverse moduli decrease as temperature increases.

Generally, the modulus of polymer matrix decreases slowly until the temperature is elevated to the glass transition point, and drops very rapidly after that point. The glass transition temperature of 350°F cure epoxy is around 250°F; so, the slope variation of the transverse and shear moduli near 250°F can be explained by thermal characteristics of the epoxy resin.

The shear moduli obtained using rigid tabs were compared with those from flexible tabs. Even though shear moduli have about 10% scatter range, there was no apparent difference between the shear moduli obtained from the conventional rigid tab and shear moduli obtained from the flexible tab. This means that the stress concentration near end tabs does not affect the shear modulus measurement of an 8.0 inch specimen.

Figure 7 shows that the Poisson's ratio decreases slightly as temperature increases. Similar results were reported by Garber et al [6] for Celion 6000/PMR-15 graphite/polyimide composites.

Strength

As shown in Figs. 6-a, b, and c, the effect of temperature on strength is similar to the case of modulus under the temperature range of 350°F. These results may also be explained by the characteristics of fiber and matrix for temperature variation. At 500°F the strength dropped to about 60% of the strength at room temperature. This may be from the decomposition of epoxy matrix at temperatures over 350°F. If the matrix is decomposed, it cannot transmit load between fibers. Since uniform stress at the cross section cannot be secured, the strength is decreased.

Comparing the shear strengths obtained from the rigid tab specimen and the flexible tab specimen in Table 3 and Figs. 6-b and c, we note that shear strengths from the solid tab are 13-30% less than strength from the flexible tabs. As discussed before, it is evident that the lower strength of rigid tab specimens is from the stress concentration near end tabs. The room temperature shear strength of 15° off-axis specimens using flexible tabs was unattainable because flexible tabs cannot transmit enough load (2200-2500 lbs) to break the specimen.

Considering the difference of shear strengths from the test using rigid tabs and the test using flexible tabs, we see that the off-axis test using the flexible fiberglas-knit/silicon-rubber tab is more suitable for shear strength evaluation.

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Table 1-a. Apparent Elastic Moduli and Strengths of 0° and 90° Off-axis Coupon Specimens

Specimen	Temp	Properties		
		Tensile Modulus (x10^6 psi)	Strength (ksi)	Poisson's Ratio
0 Degree	75°F	20.80	294.60	0.30
		20.70	283.70	0.31
	250°F	21.80	313.50	0.29
		21.30	288.30	0.28
	350°F	21.10	266.60	0.28
		21.50	295.20	0.29
	500°F	19.50	171.00	
		21.00	162.00	
90 Degrees	75°F	1.33	9.82	
		1.32	8.15	
		1.40	9.79	
		1.47	7.43	
	250°	1.13	7.02	
		1.17	6.02	
		1.17	6.43	
	350°	0.13	1.55	
		0.16	2.25	
		0.12	1.36	

Table 1-b. Apparent Elastic Moduli and Strengths of 15° and 45° Off-axis Coupon Specimens

Specimen	Temp	Properties Tab	Tensile Modulus (x10^6 psi)	Strength (ksi)
15 Degrees	75°F	Solid Tab	9.00	37.20
			8.95	39.30
		Flex. Tab	8.60	-
			8.52	-
	250°F	Solid Tab	7.30	20.10
		Flex. Tab	7.40	24.70
			7.23	26.20
	350°F	Solid Tab	0.99	7.58
		Flex. Tab	0.91	10.47
			0.98	11.80
45 Degrees	75°F	Solid Tab	2.05	11.60
			1.73	12.55
		Flex. Tab	1.91	13.20
			1.91	12.80
	250°F	Solid Tab	1.44	9.05
		Flex. Tab	1.58	9.40
			1.52	9.94
	350°F	Solid Tab	0.18	2.11
		Flex. Tab	0.17	2.56
			0.13	2.56

Table 2. Longitudinal, Transverse and Shear Moduli for Temperature Variation

Temp. / Modulus ($x10^6$ psi)			75°F	250°F	350°F	500°F
E_1			20.80	21.60	21.30	20.30
E_2			1.38	1.16	0.14	-
G_{12}	From 15° Coupon	Solid Tab	0.92	0.66	0.067	
		Flex. Tab	0.85	0.66	0.064	
	From 45° Coupon	Solid Tab	0.73	0.53	0.066	
		Flex. Tab	0.74	0.58	0.051	
ν_{12}			0.31	0.29	0.28	

Table 3. Longitudinal, Transverse and Shear Strength for Temperature Variation

Temp. Strength (ksi)			75°F	250°F	350°F	500°F
X			298.20	300.20	280.90	166.50
Y			8.80	6.51	1.72	
S	From 15° Coupon	Solid Tab	10.10	5.15	1.98	
		Flex. Tab	-	6.63	3.18	
		% Diff.	-	22.3%	37.7%	
	From 45° Coupon	Solid Tab	8.31	6.29	1.33	
		Flex. Tab	9.64	7.22	1.92	
		% Diff.	13.8%	12.9%	30.7%	

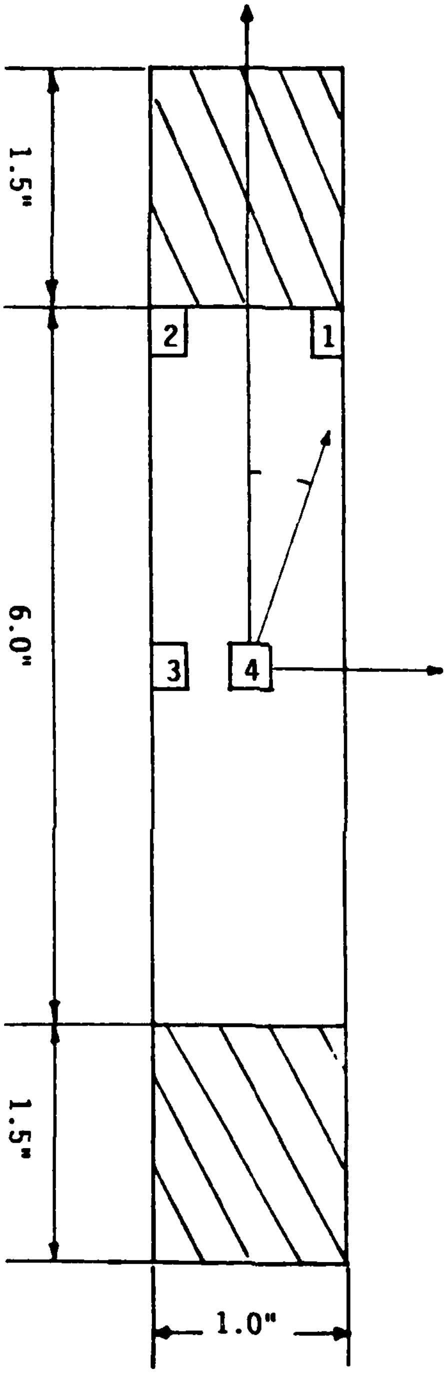

Fig. 1. Specimen geometry and positions of strain gages

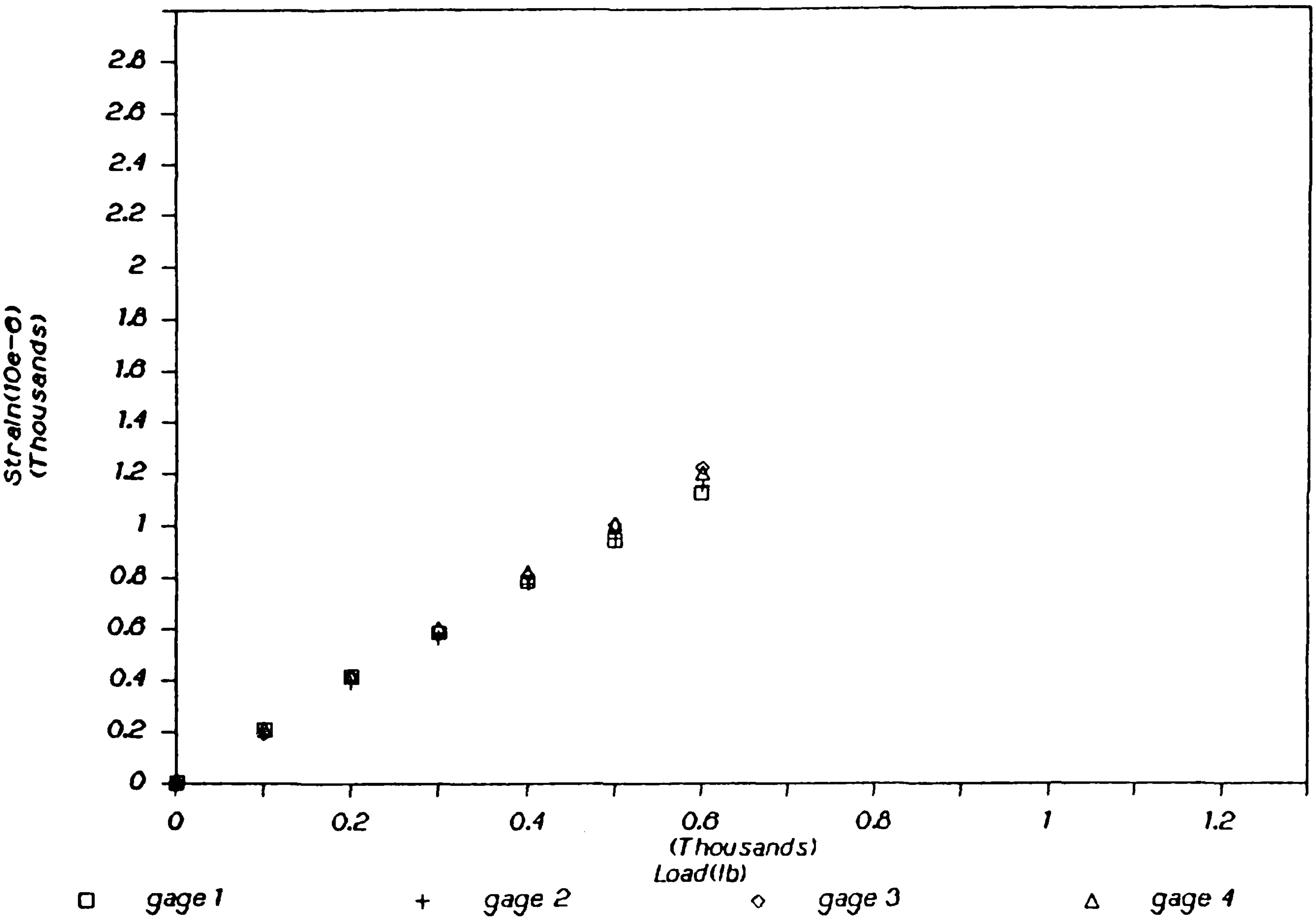

Fig. 2-a. Strains at four locations in a 10° specimen using the new fiberglas-knit/silicone-rubber tabs (grip pressure: 400 psi)

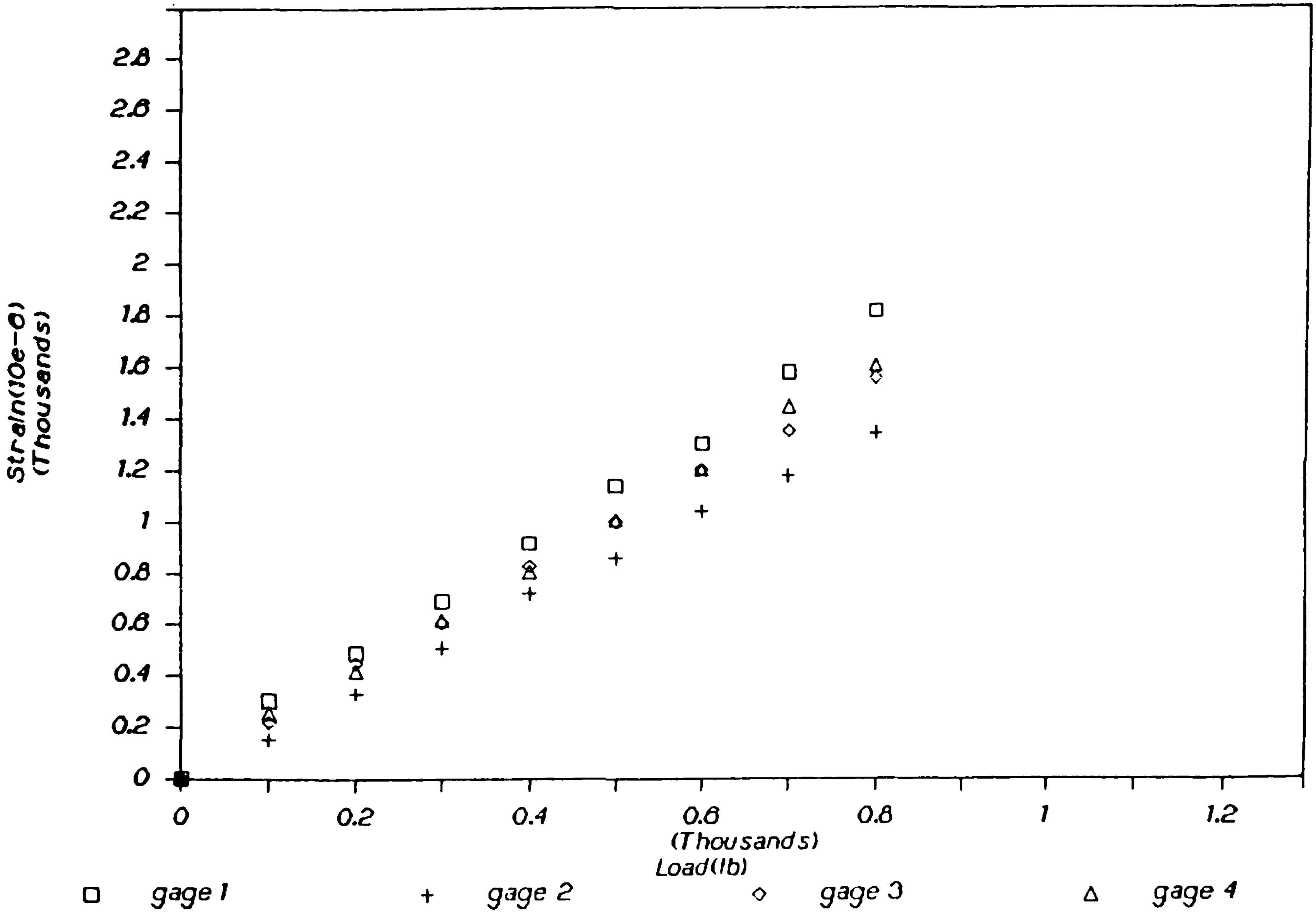

Fig. 2-b. Strains at four locations in a 10° specimen using conventional glass/epoxy end tabs (grip pressure: 400 psi)

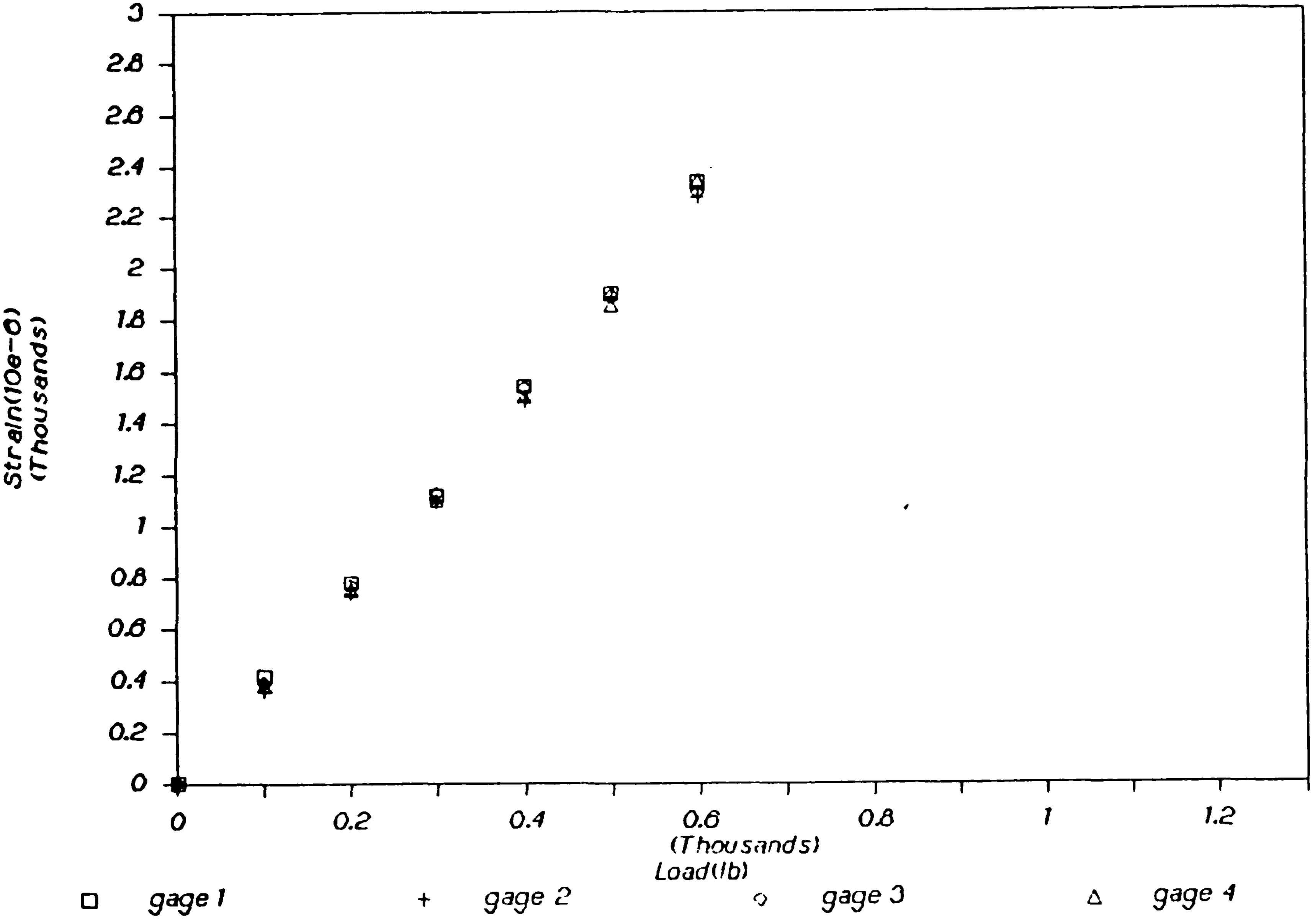

Fig. 2-c. Strains at four locations in a 20° specimen using the new fiberglas-knit/siliconerubber tabs (grip pressure: 400 psi)

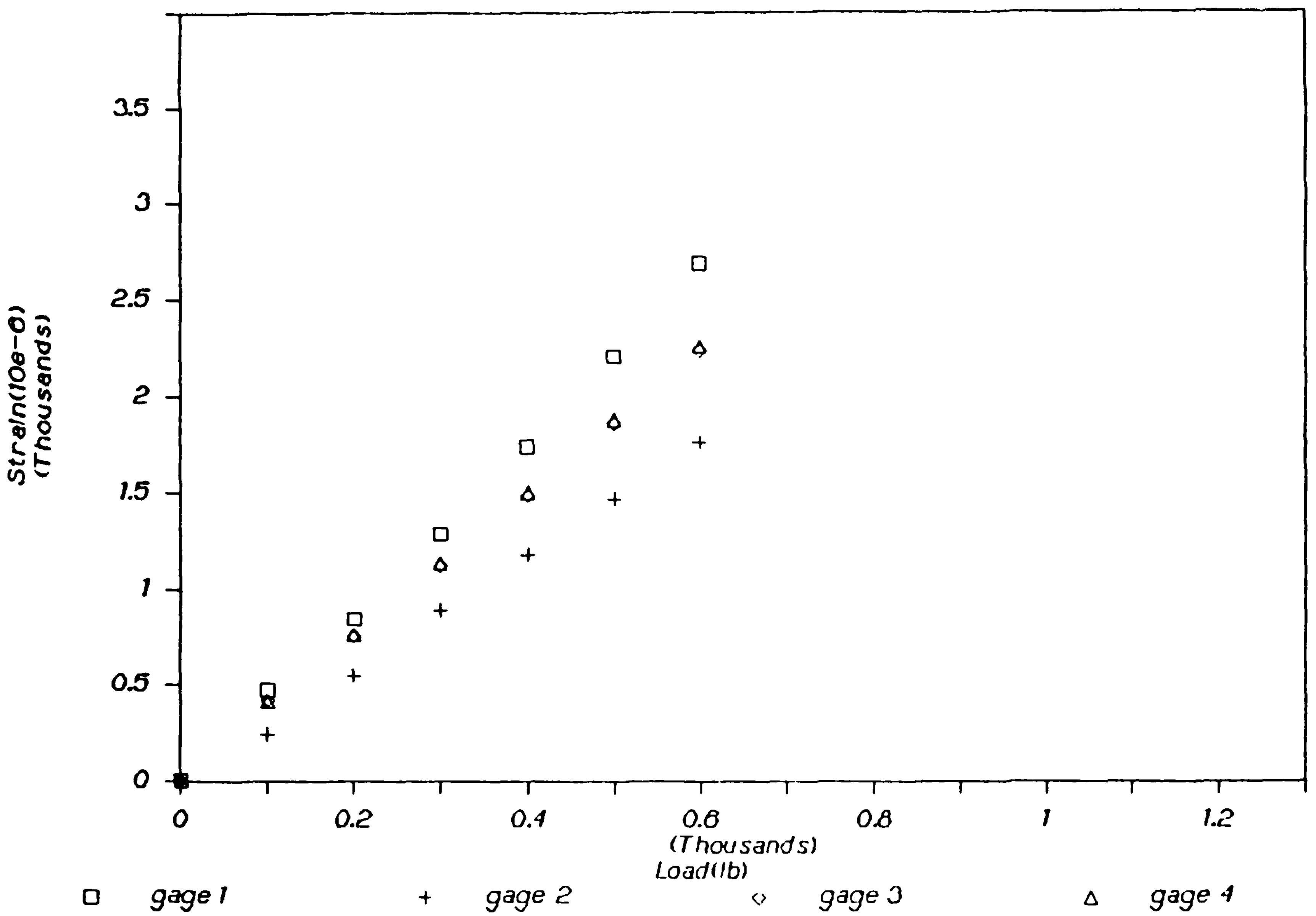

Fig. 2-d. Strains at four locations in a 20° specimen using conventional glass/epoxy end tabs (grip pressure: 400 psi)

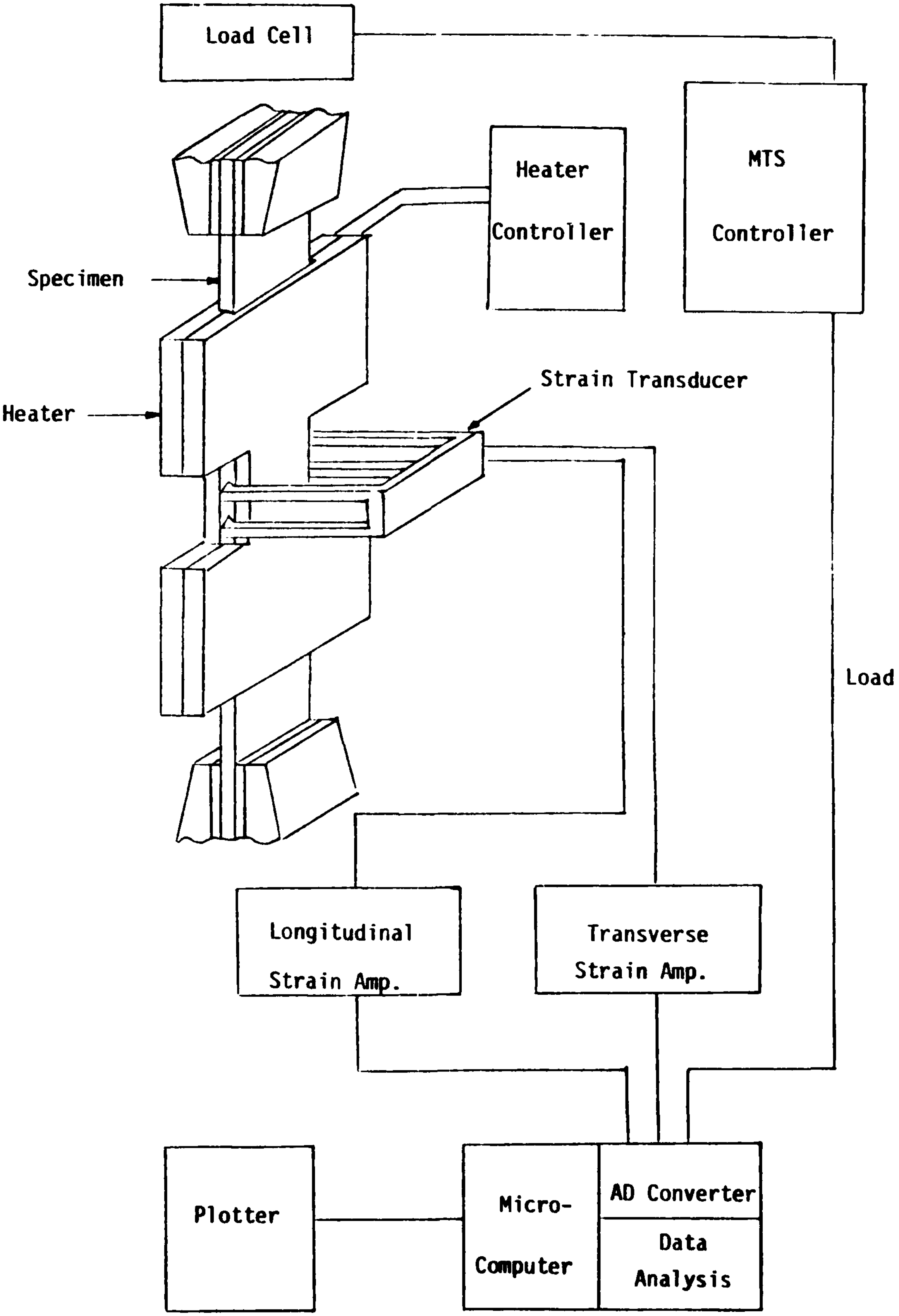

Fig. 3. Schematic for testing system

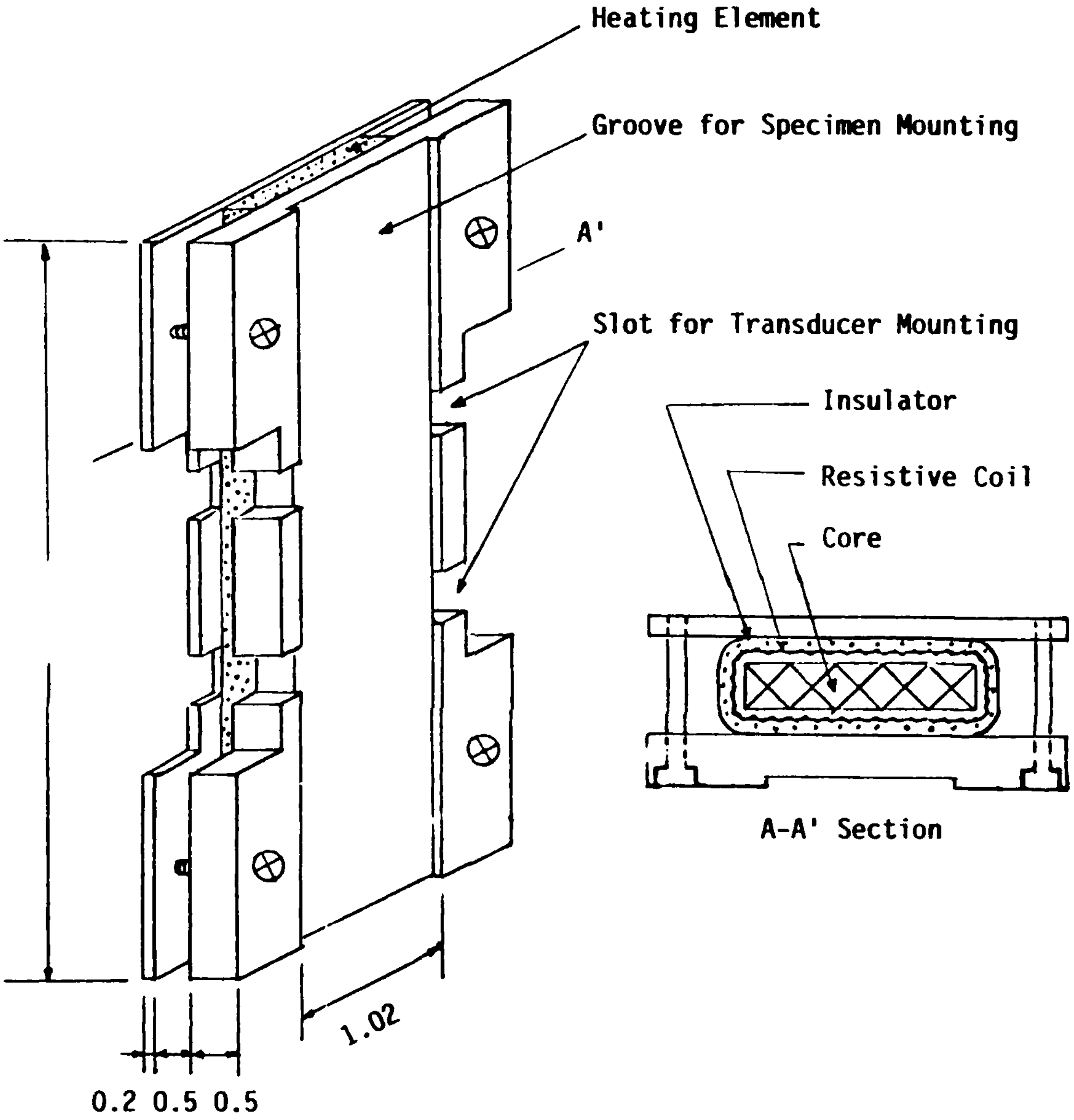

Fig. 4. Schematic for heating plate

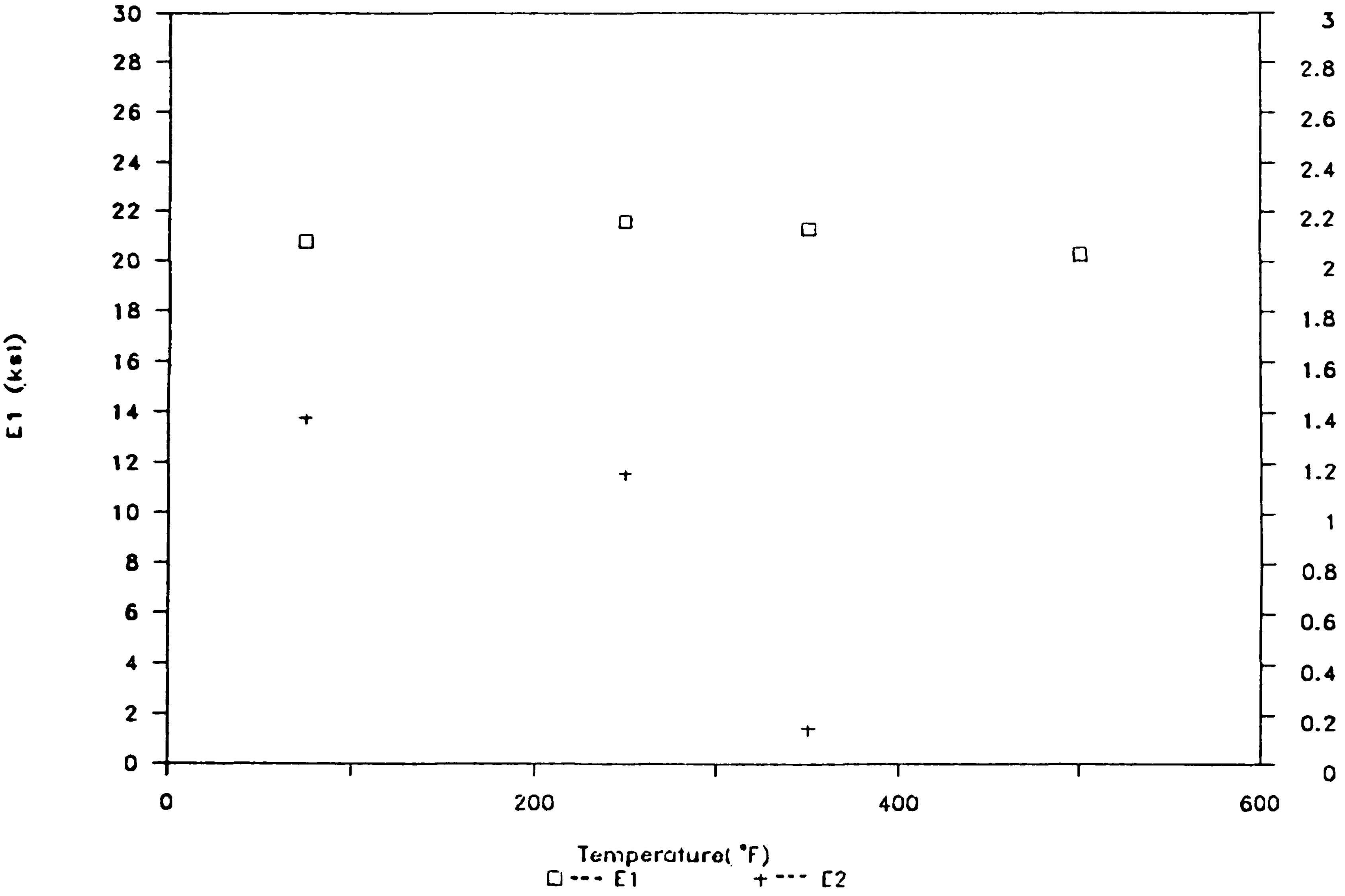

Fig. 5-a. Longitudinal and transverse modulus vs. temperature

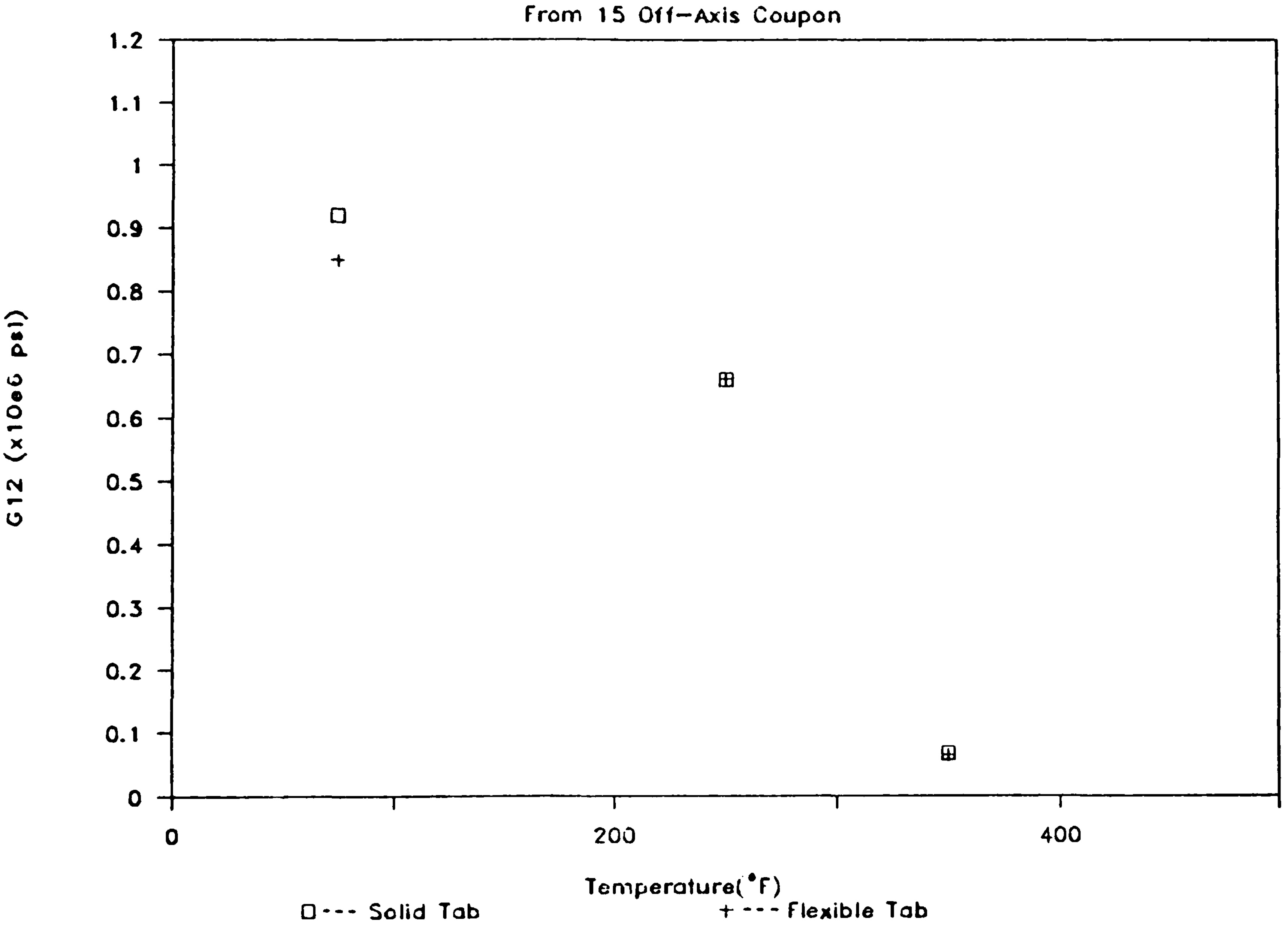

Fig. 5-b. Shear modulus vs. temperature obtained from 15° off-axis coupon specimen

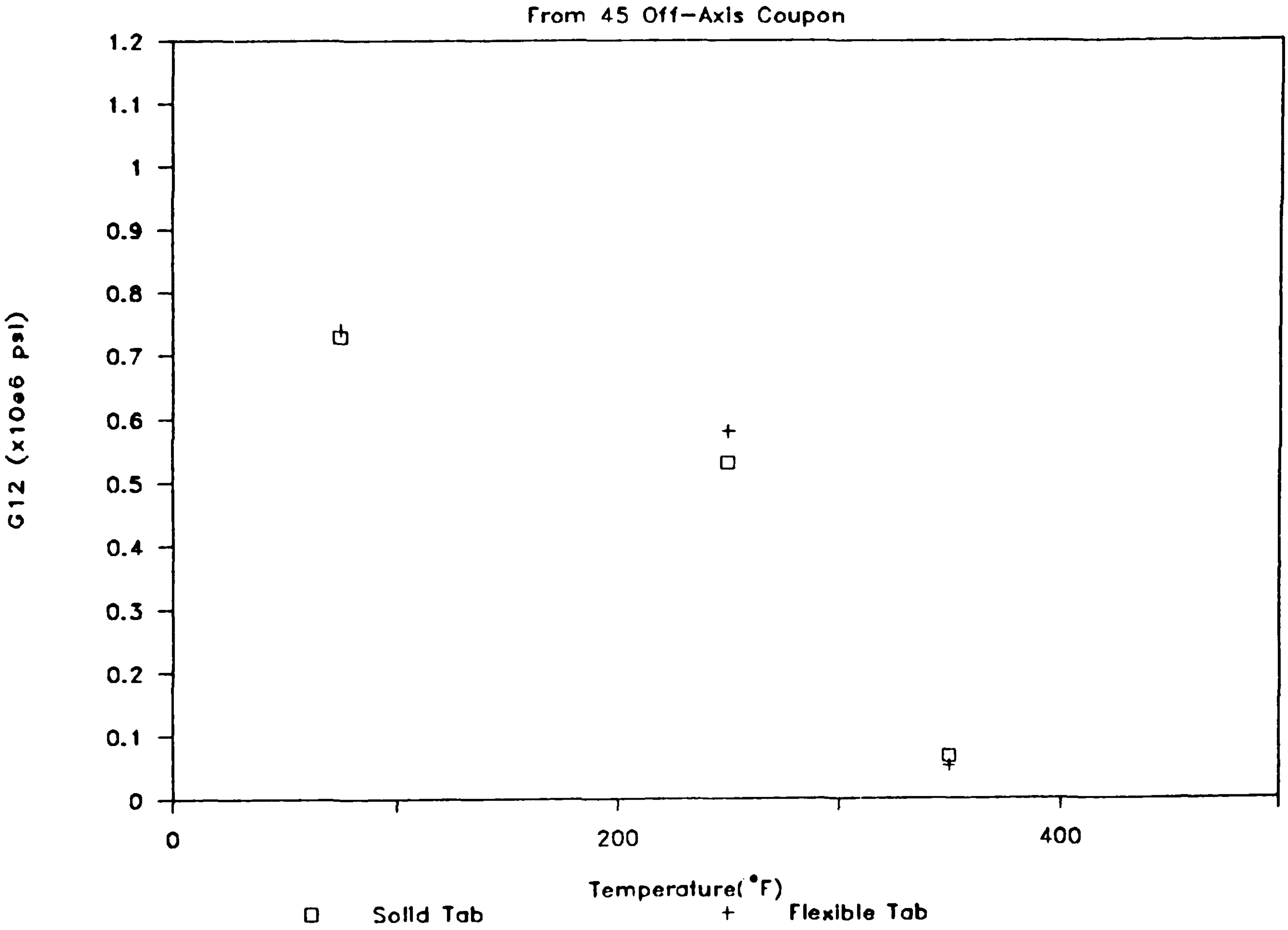

Fig. 5-c. Shear modulus vs. temperature obtained from 45° off-axis coupon specimen

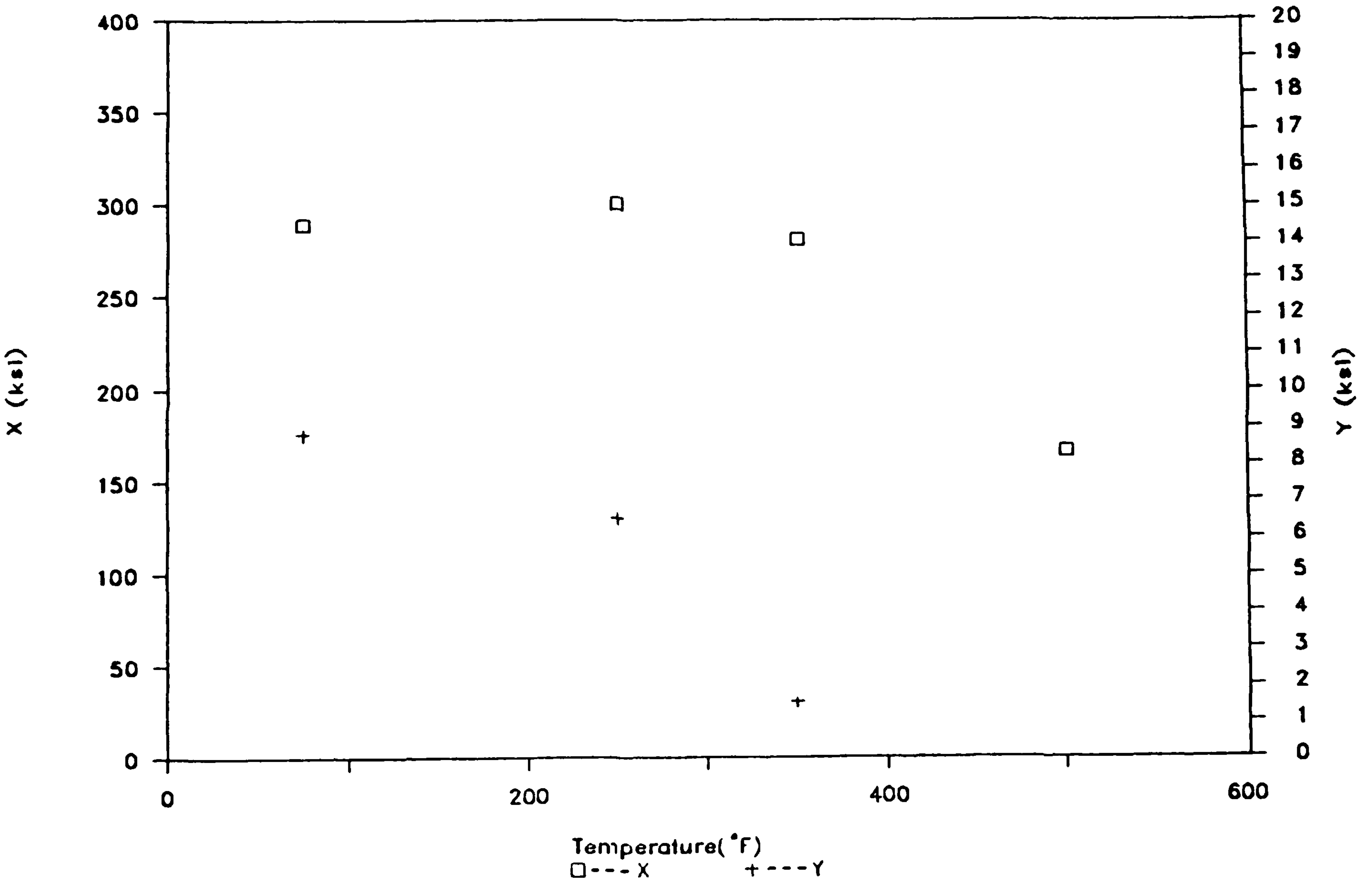

Fig. 6-a. Longitudinal and transverse strength vs. temperature

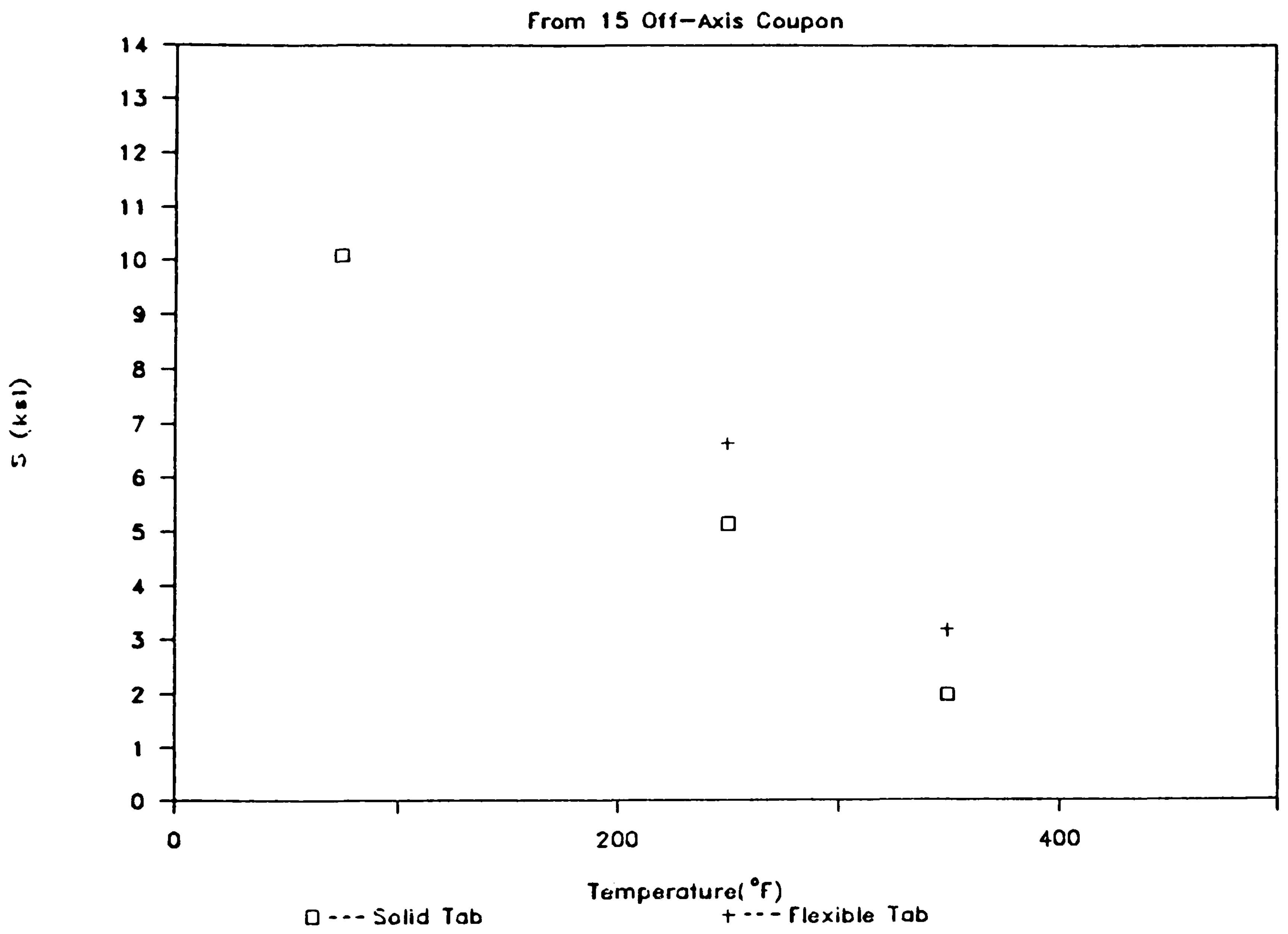

Fig. 6-b. Shear strength vs temperature obtained from 15° off-axis coupon specimen.

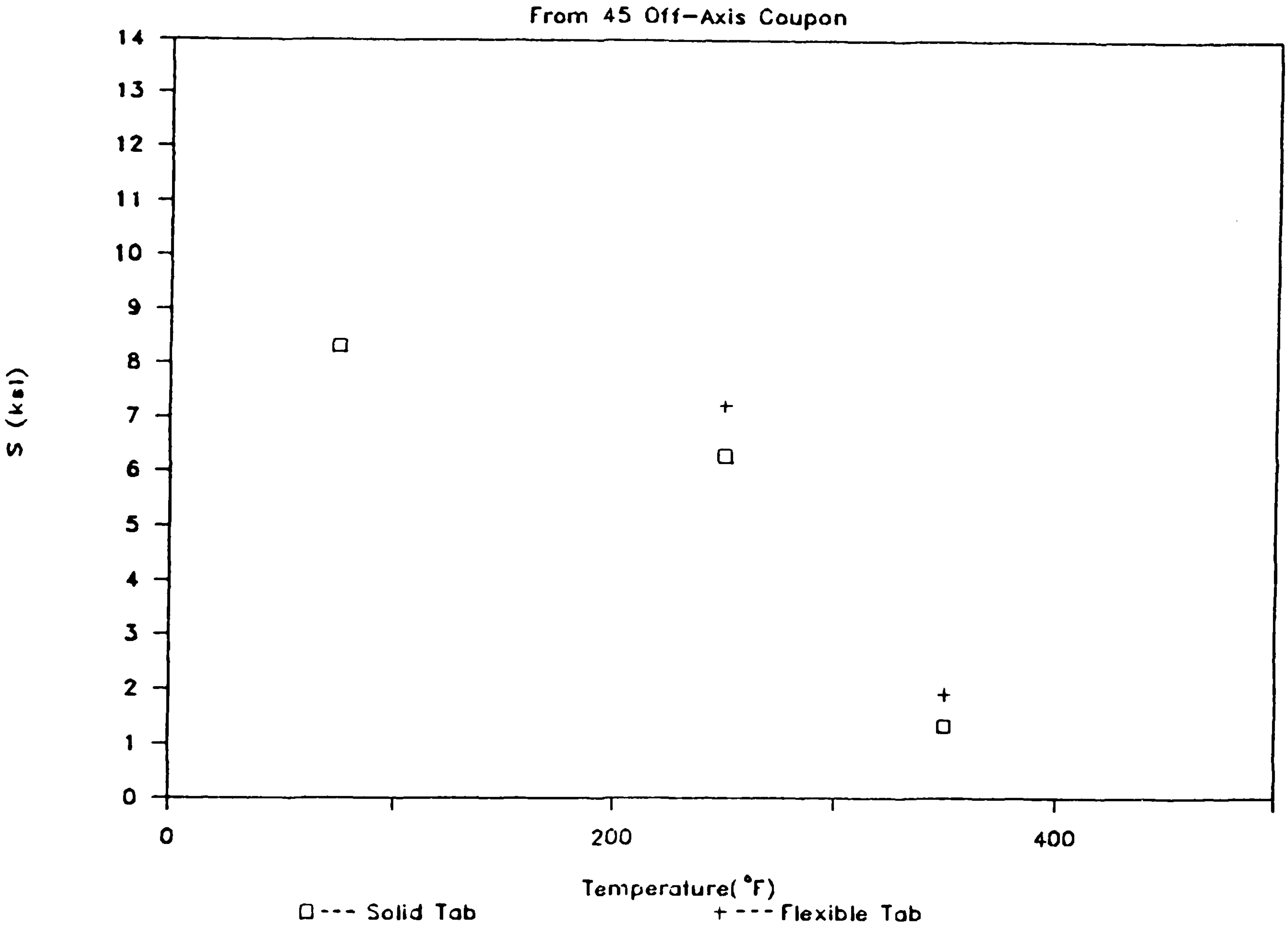

Fig. 6-c. Shear strength vs. temperature obtained from 45° off-axis coupon specimen

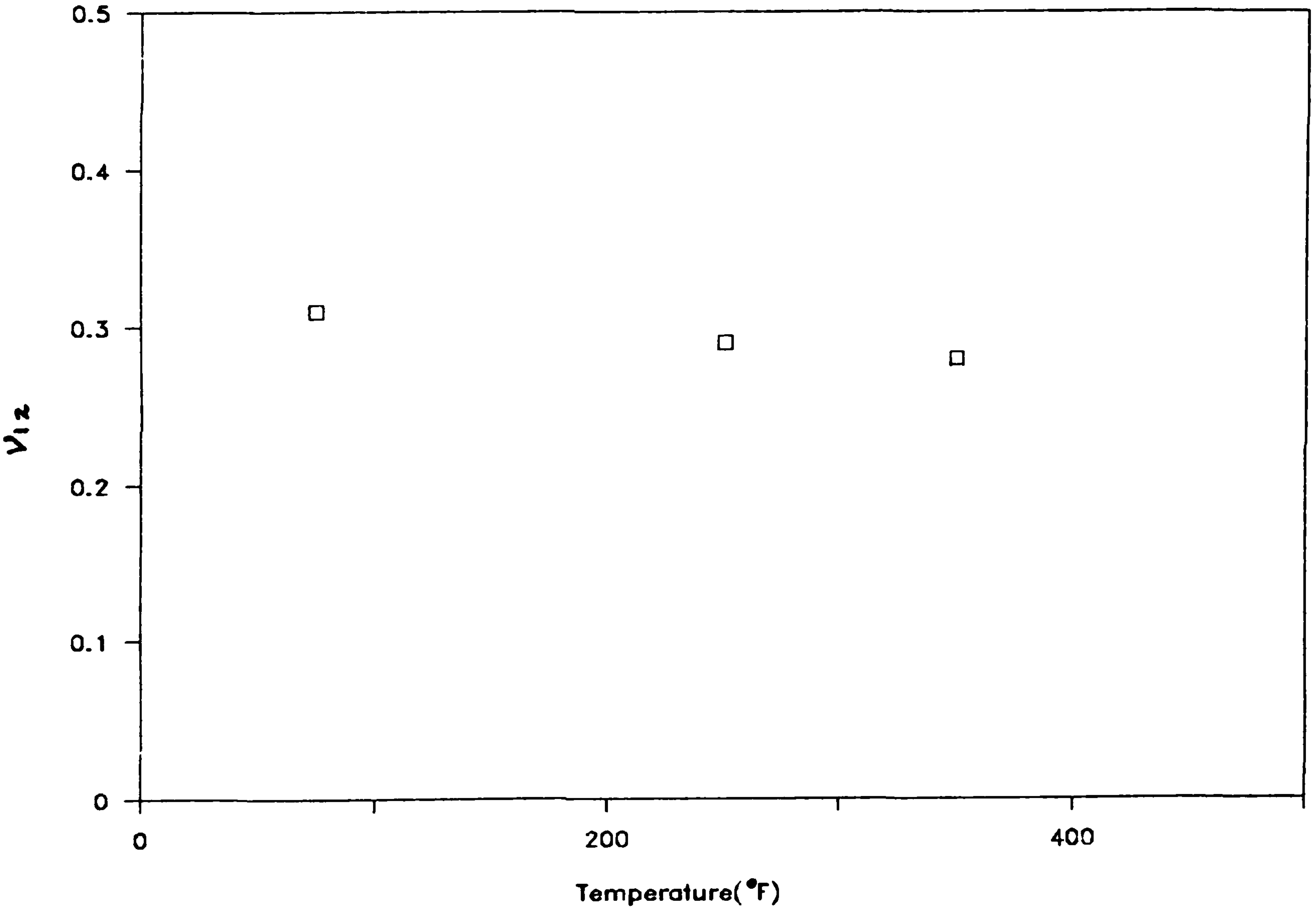

Fig. 7. Poisson's ratio vs. temperature