COO-1794-7

CYCLIC FATIGUE OF BRITTLE MATERIALS

PROGRESS REPORT

MASTER

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I. OBJECTIVES OF RESEARCH

The objective of this study has been to investigate the fatigue behavior (static and cyclic) of brittle materials subjected to uniaxial and combined stresses, primarily torsion. The studies of alumina and graphite are presented in report COO-1794-6. Therefore, this progress report will only summarize the research thus far accomplished on the brittle polymers. The objective here has been to study the flexural fatigue characteristics of epoxy (Epon 828) and polyester (Paraplex P43) resins. A rotating beam fatigue machine which maintains a constant stress and a plate bending fatigue machine which maintains a constant displacement are both being used for this investigation. These methods provide a zero mean stress with alternating tension and compression stresses. The effect of cyclic frequency is being investigated as well as the effect of the environment, particularly, the role of water vapor. Temperature rise during fatigue testing is measured using an infra-red radiation thermometer which does not contact the specimen. The strength, modulus and hysteresis are then studied as a function of temperature to determine how the temperature rise during fatigue will influence the ultimate failure. Fracture surfaces are studied by optical, interference and electron microscopy to relate the crack growth to the fatigue experiment.

II. SELECTION OF MATERIALS

The two polymeric materials selected to study the fatigue characteristics of brittle materials were an epoxy, Epon 828+, and a polyester, Paraplex P-43*. The flexural properties for these materials as determined by three point bending tests are as follows.

1. Epon 828 (cured with 14.6% by weight of Meta phenylene diamine)

Flexural strength	20,400 psi
Flexural modulus	4.1×10^5 psi
Ultimate strain	6.6%

2. Paraplex P-43 (cured) with 1% by weight of Benzoyl Peroxide)

Flexural strength	15,400 psi
Flexural modulus	6.14×10^5 psi
Ultimate strain	2.75%

III. PREPARATION OF MATERIALS

The procedure suggested by the materials manufacturer was followed in preparation of the mixture for casting of specimens. For the epoxy resin (Epon 828), meta phenylenediamine (MPDA) curing agent was used. The mixing procedure was as follows:

i) 15 parts (w) of Epon 828 were heated to 150°F.

ii) This was then mixed with the 14.6 parts per hundred of molten (150°F) MPDA curing agent.

iii) The above mixture was then mixed with the remaining 85 parts (w) of Epon 828 at room

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+ Tradename, Shell Chemical Co.
* Tradename, Rohm & Haas Co.

temperature. The mixing was done under vacuum to minimize the entrapment of air bubbles in the casting mixture.

The casting mixture was then spun in a centrifuge at 1500 rpm for 30 minutes. This was done to remove the minute air bubbles which are entrapped in the resin during the mixing operation. The resin was then poured into cylindrical Teflon molds preheated to 200°F. The molds were preheated to reduce the viscosity of the resin at the walls of the mold to obtain good wetting. The casting mixture was poured very slowly into the molds to avoid the entrapment of air bubbles in the molds.

The molds were then maintained at room temperature for 8 hours until a solid gel was formed. They were then placed in an oven at 200°F for 45 minutes followed by a post cure at 350°F for 1 hour.

The components used to cast the polyester specimens were as follows:

Paraplex P-43	100 parts by weight
Styrene monomer	7 parts by weight
Benzoyl Peroxide	1 part by weight

The one part of Benzoyl Peroxide was dissolved in 7 parts of styrene monomer and was then mixed with 100 parts of resin. The mixture was spun in a centrifuge at 1500 rpm for 3 minutes and then poured into the Teflon molds similar to the epoxy resin procedure. The molds were maintained at 75°F for 6 to 8 hours to allow for the escape of any air bubbles entrapped during the casting operation.

The molds were then placed in the oven at 150^{o}F for one hour followed by 230^{o}F for another one hour.

IV. PREPARATION OF SPECIMENS

The cast bars which were 11/16" in diameter and 5-1/2" long were machined to the final shape of the specimens. The machining was done on a lathe with a profile cutting attachment. The specimens were then polished using wax polishing compounds. The specimen used for fatigue testing on the rotating beam fatigue machines is shown in Fig. 1. Specimens were also prepared for flexural tests and for fatigue studies using a plate bending fatigue machine.

For the preparation of these specimens, the resin was cast in the form of a flat plate using an aluminum mold. The mold was placed between the platens of a hydraulic compression press under slight pressure. The curing cycle was the same as described earlier.

For machining these fatigue specimens a special template attachment was designed and used on a tensilkut router. Specimens used for 3 point bending tests, were cut from the cast plate using a diamond plated cutting wheel. These specimens are shown in Fig. 1.

V. EXPERIMENTAL PROCEDURES

The Budd Co. model RBF25 fatigue machine was used for the rotating beam fatigue tests. This machine has an adjustable spindle speed (5000-12000 cpm) and is provided with a cyclic counter (999,999,900 maximum count). It has a calibrated beam and poise system which can apply a moment up to 25 in. -lb. to the cantilevered end of the specimen. The machine is provided with a micro switch which is actuated when the specimen breaks and thus shuts off the machine and cyclic counter.

Before starting the actual test, the drive spindle collet with specimen was rotated manually to check for run out at the free end of the specimen. The specimens having an excessive run out ($\geq$.004) at the free end were not used for the fatigue testing.

The specimen was loaded after the machine was brought up to the operating speed. The cycle counter was then adjusted to zero reading. The operating speed was again checked and corrected for any reduction in speed due to the application of load.

A plate bending fatigue machine is also being used. The plate bending fatigue machine is of the fixed-cantilever, repeated constant deflection type. In this machine the specimen is held as a cantilever beam in a vise at one end and bent by a concentrated load applied to a holder fastened to the other end. The bending is accomplished by a connecting rod driven by a variable eccentric mounted on a shaft. This shaft is rotated at constant speed by a motor. The vise may be set in the plane of the eccentric so that the beam is deflected the same amount on either side of the neutral position (completely reversed stress) or the vise may be set so that the deflection is greater on one side than on the other (mean stress not zero). In this machine the specimens are not rotated as is the case of the rotating beam fatigue machine. The plate bending fatigue machine produces a constant amplitude of deflection in the test specimen each cycle whereas the rotating beam fatigue machine produces a constant amplitude of force on the test specimen each cycle.

An IRCON Radiation thermometer (infra-red) CH-34L series was used to measure the surface temperature of the specimen during the fatigue testing.

This radiation thermometer operates in a very narrow spectral band centered at 3.43 microns coincident with the carbon-hydrogen absorption band. The temperature measurements are made without contacting the object by sensing the infra-red radiation emitted from the surface. This instrument has five temperature spans and its repeatability is 2°F above 120°F and 4°F below 120°F. The focussing range of the optical head is 7 in. to infinity and the minimum target size which can be focussed on is a 0.155" diameter circle.

The emittance of the material whose temperature is to be measured must be known in order to obtain accurate temperature readings. The emittance values for epoxy and polyster resins were not available and as such it was necessary to establish this for both the materials experimentally.

A specimen was first coated with 'SPRAY-GRAPH'* over a small region of the surface. After having applied a thin but visibly opaque coating, the specimen was heated to 260°F by using a hot air blower. The optical head of the thermometer was focussed on the coated surface patch and the emmittance

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*Trade mark for a graphite coating.

dial was set to 0.8 which is the known emittance of the graphite coating. The true graphite temperature was then read on the temperature scale of the instrument. Next, the uncoated portion of the specimen immediately adjacent to the coated area was focussed on and the emittance dial was rotated to obtain the same temperature reading as before. This established the emittance value for the material under consideration.

VI. RESULTS

The flexural (3 point bending) stress-strain behavior of both the epoxy and polyester resins was determined at various temperatures since temperature increases occurred during the fatigue cycling. Three different cross-head rates were used but the results for only one of the rates are shown in Figs. 2 and 3. The flexural strengths for both the polyester and epoxy resins are plotted versus temperature in Fig. 4. The flexural modulus of elasticity for both resins is plotted versus temperature in Fig. 5. It can be seen that the strength and modulus of the polyester resin are greatly reduced as the temperature is increased to 200^{o}F. Thus, if the temperature of the material is increased during the fatigue test, the life of the specimen will be determined by the property changes occurring due to the temperature increase.

The temperature change during fatigue cycling has been determined using the infra-red radiometer as described in the previous section. The temperature rise is being studied as a function of cyclic frequency and stress level. Some of the results obtained thus far are shown in Figs. 6 through 9. The surface temperature for the epoxy resin at a cyclic frequency of 4400 cycles/minute is shown in Fig. 6 for two different stress levels. Surface temperatures up to 190°F are measured at the time of specimen fracture. The effect of frequency is shown in Fig. 7 and it can be observed that the maximum surface temperature is increased with increasing frequency. The results for the polyester resin at various stress levels are shown in Fig. 8 and the results are somewhat different from those observed for epoxy resins. For the polyester, a very rapid temperature increase is observed in the last several cycles of its life. The results from Fig. 8 are replotted in Fig. 9 as a function of time rather than the log of number of cycles.

The actual fatigue results thus far obtained with the rotating beam fatigue machine are presented in Figs. 10-12. At least 100 specimens have been tested to establish these curves. For the epoxy resin, only a small stress range can be evaluated because of the low modulus of the material and the resulting large deflection of the specimen when loaded. Thus, an upper limit is placed on the stress which can be applied to the specimen. Figs. 10 and 11 are fatigue curves showing in detail the failure stress versus the number of cycles to failure. Fig. 12 includes the single cycle strength and shows the results for both polyester and epoxy resins. Endurance limits exist for both materials.

Studies have also been initiated on the effect of the environment on cyclic fatigue of these materials. One method employed to isolate the specimens from water vapor in the atmosphere was to coat the specimens before they were fatigued. The epoxy specimens and polyester specimens were held at 100°C and

50°C respectively for 2 hours and then coated with a graphite coating. The coating was done in the oven itself so that specimens could not absorb any water from the atmosphere during the coating operation. Finally a silicone wax was applied to the surface over the graphite coating. These specimens were then cycled at one stress level and frequency and the number of cycles to failure compared to uncoated specimens subjected to the same stress level and frequency. The probability of failure is plotted versus the number of cycles to failure for the epoxy and polyester specimens in Figs. 13 and 14, respectively. The life of the epoxy specimens is increased by the coating while the polyester specimens are unaffected. However, the polyester specimens could only be heated to 50°C, as they softened too much above this temperature and this is not sufficient to drive off water absorbed in the specimen or absorbed on the surface.

Experiments are also being conducted to assess the affect of cycling the specimens while immersed in water. A chamber is placed around the specimen in the rotating beam fatigue machine and water is circulated through the chamber. The composite affect of the water is to not only provide an atmosphere for the specimen but it also acts to reduce the temperature of the specimen. The result is an increase in fatigue life as shown by the points in Fig. 11. Further studies are being conducted in order to clarify the phenomenom.

Studies are also being conducted of the fracture surfaces. Both optical and interference microscopy are being utilized and it is planned to investigate portions of the surface with the electron microscope.

Figure 15 shows fracture surfaces of fatigued epoxy specimens. The crack starts at the surface and can be attributed to a surface flaw produced by machining. The crack propagates radially in a stable mode until it reaches a critical radius and then propagates in an unstable mode through the specimen. It appears that the size of the mirror area seen in the photographs is dependent upon the stress applied.

Fig. 16 shows the fractured surface of the polyester specimens. The mirror area contains fatigue striations identical to the markings found on other materials subjected to fatigue and representative of cyclic crack growth. In some cases, the crack has begun at an internal flaw site which may be either a small air bubble or an inclusion (Fig. 16c).

VII. FUTURE WORK

The work to be accomplished in the remaining three months of the current contract period is described here.

Studies will be completed on delayed failure of alumina rings subjected to internal pressure. Static and cyclic fatigue studies at various frequencies will be completed on polyester and epoxy polymers. Studies on the effect of the environment will be continued in order to distinguish between the observations discussed above. In addition, the fracture surfaces will be analyzed and features of the surfaces will be related to the stress level and other parameters of the fatigue tests. Also, cumulative damage tests will be conducted to determine the effect of fatigue on the strength of the material.

VIII. COMPLIANCE WITH CONTRACT REQUIREMENTS

The principal investigator has devoted 2 summer months full time and 20% of his time during the academic year. Approximately 20% of his time will be devoted to the remainder of this current contract. Two graduate research assistants have been supported for the full year. Mr. Mahajan and Mr. Gaggar have used the results of their studies for their M.S. theses.

More emphasis than originally intended has been given to brittle polymers as opposed to inorganic glass because of better control over materials and test specimens. Also, more emphasis has been placed on static and cyclic fatigue under bending than under combined stresses in order to more thoroughly understand the phenomenom under simpler stress states.

IX. ACKNOWLEDGMENT

We gratefully acknowledge the support given to us by the Atomic Energy Commission for the pursuit of these studies.

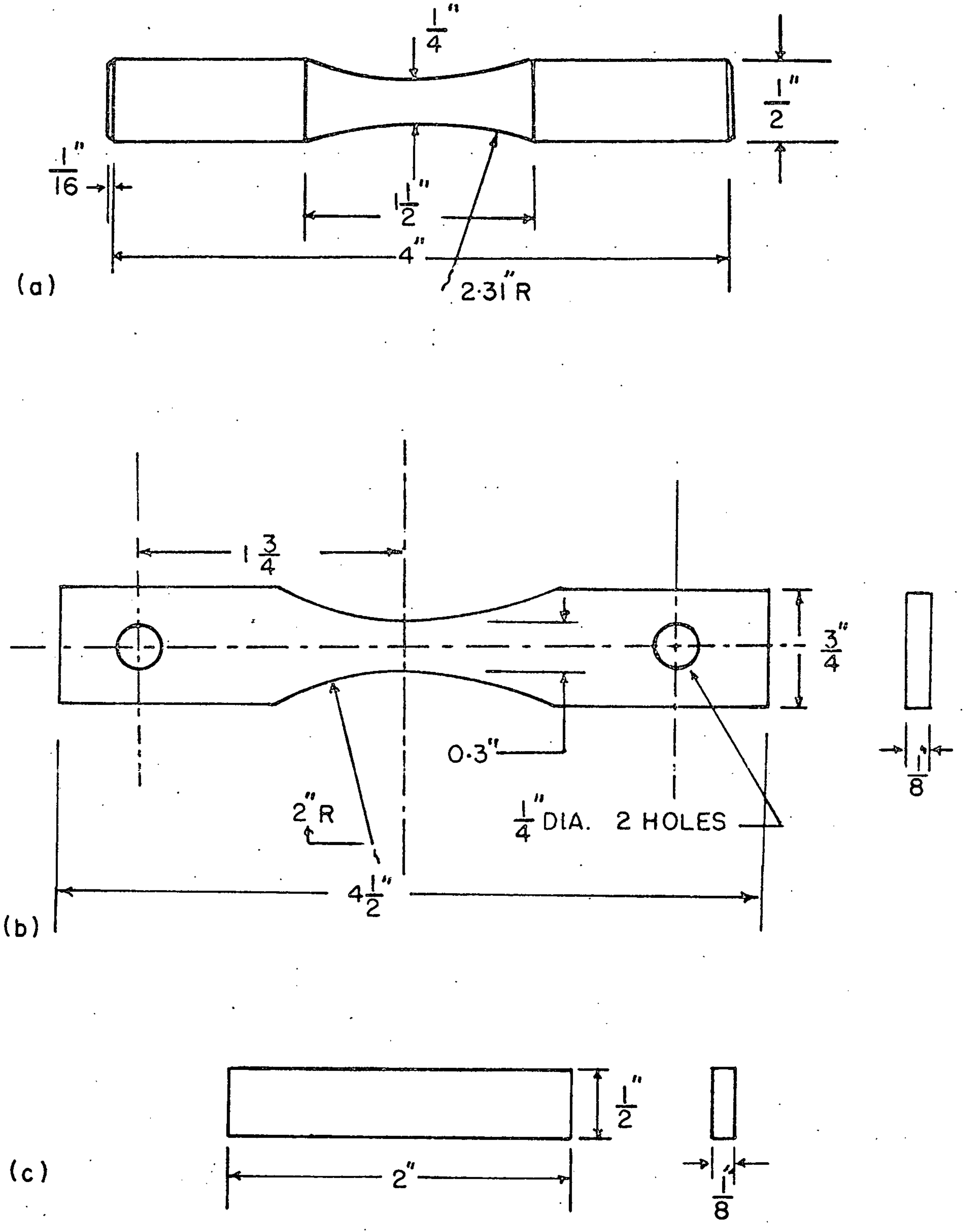


FIGURE 1 TEST SPECIMENS

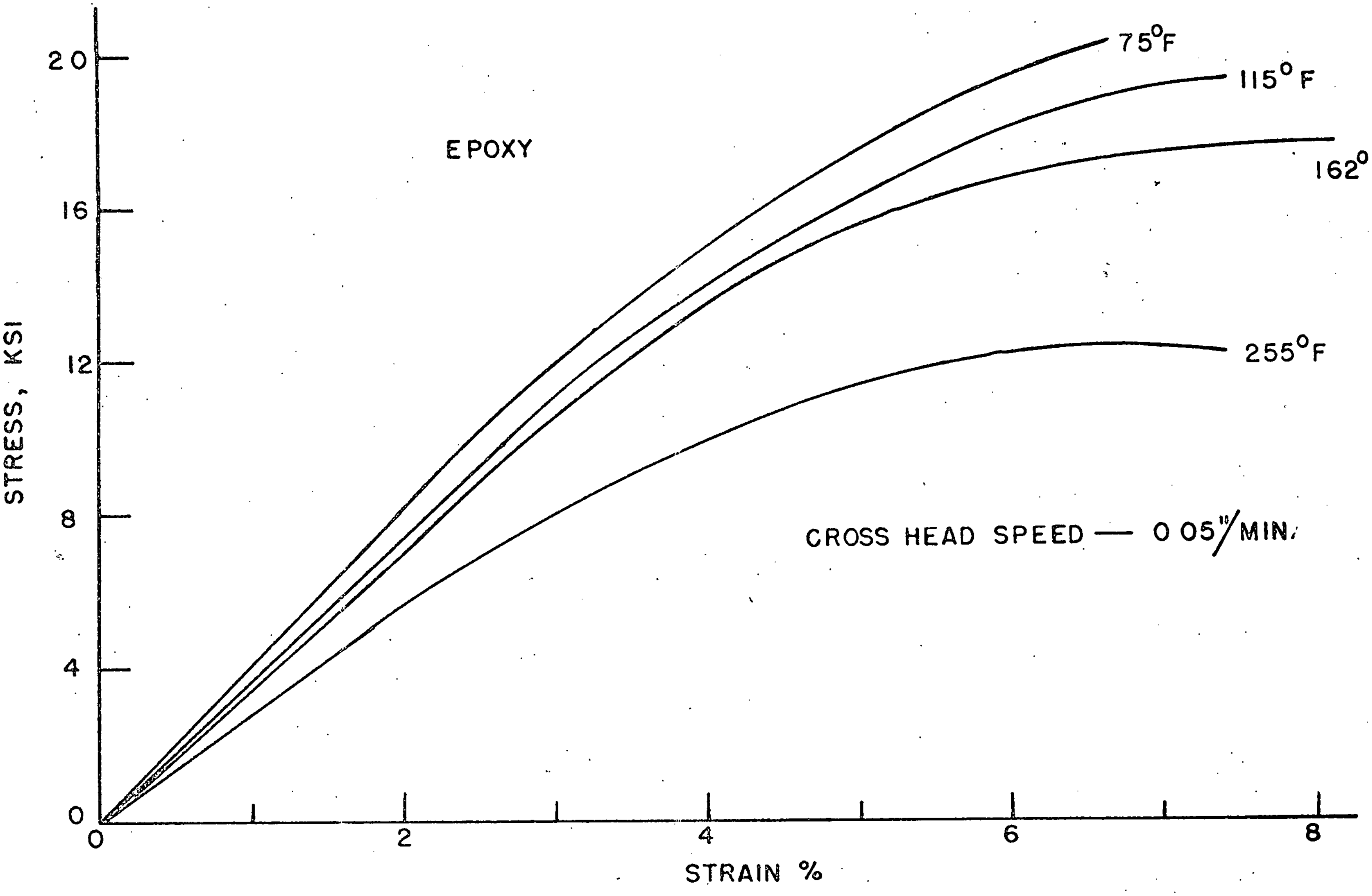


FIGURE 2. FLEXURAL STRESS-STRAIN CURVES AT VARIOUS TEMPERATURE

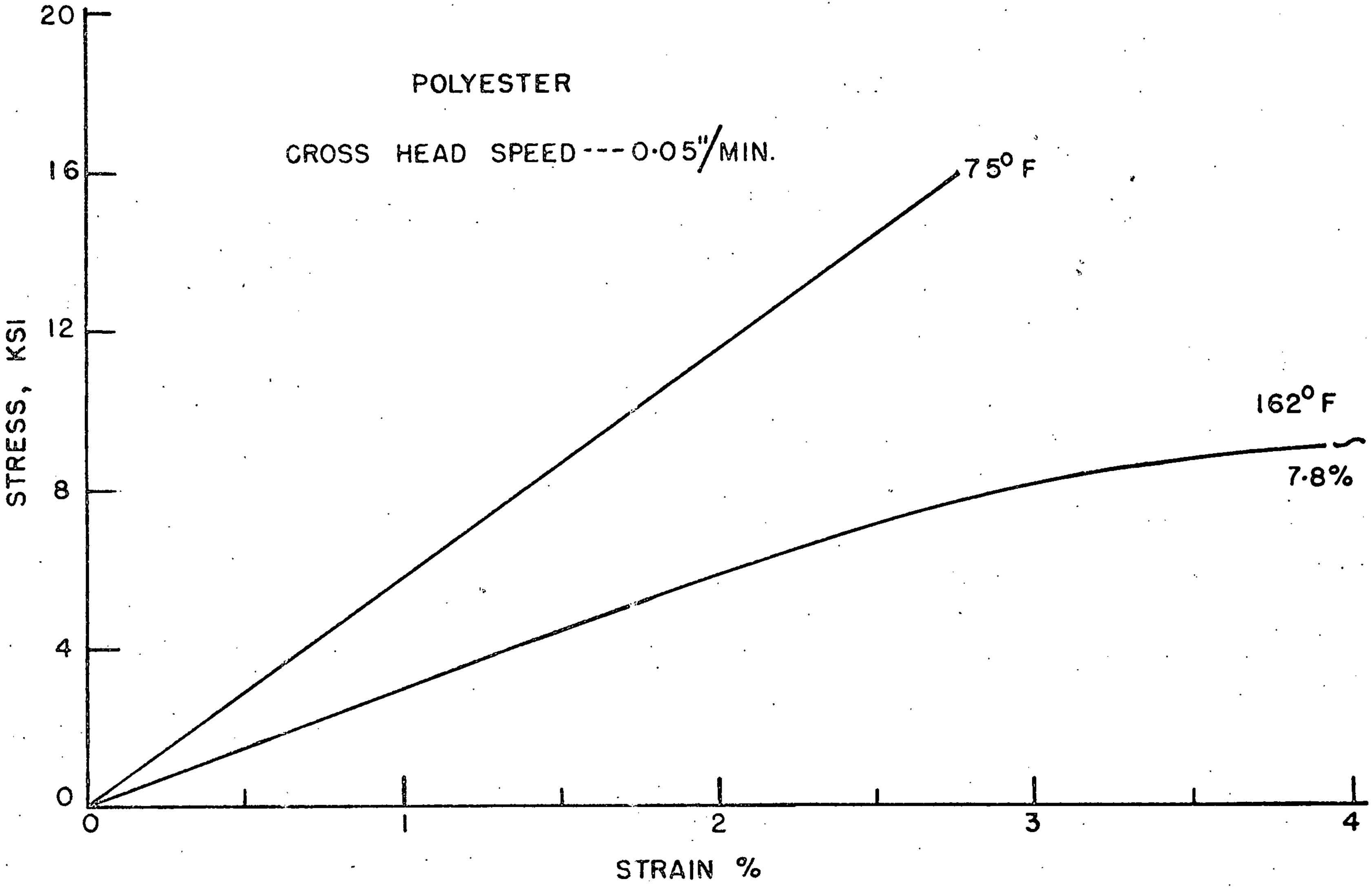


FIGURE 3. FLEXURAL STRESS-STRAIN CURVES AT VARIOUS TEMPERATURE

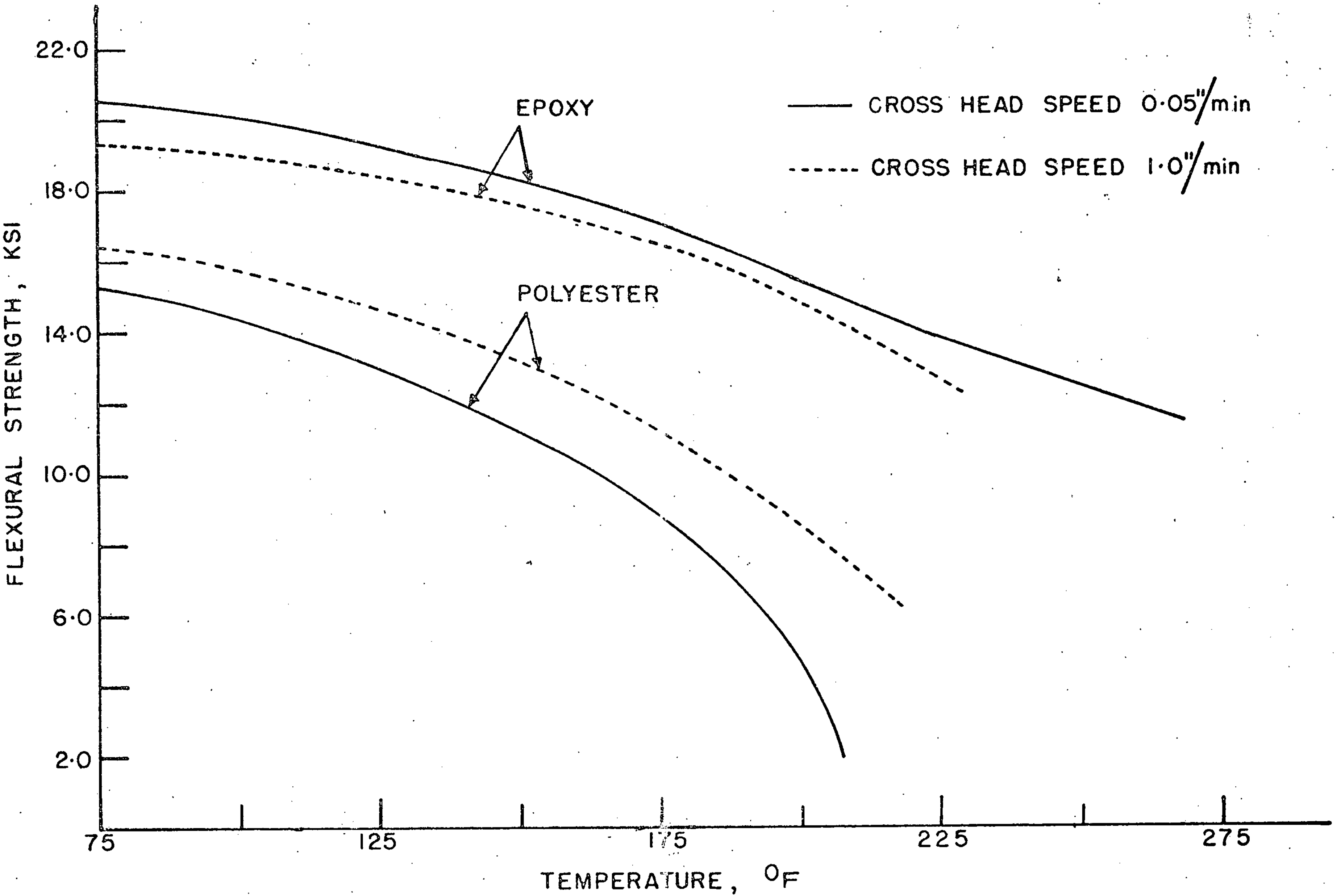


FIGURE 4. VARIATION OF FLEXURAL STRENGTH WITH TEMPERATURE

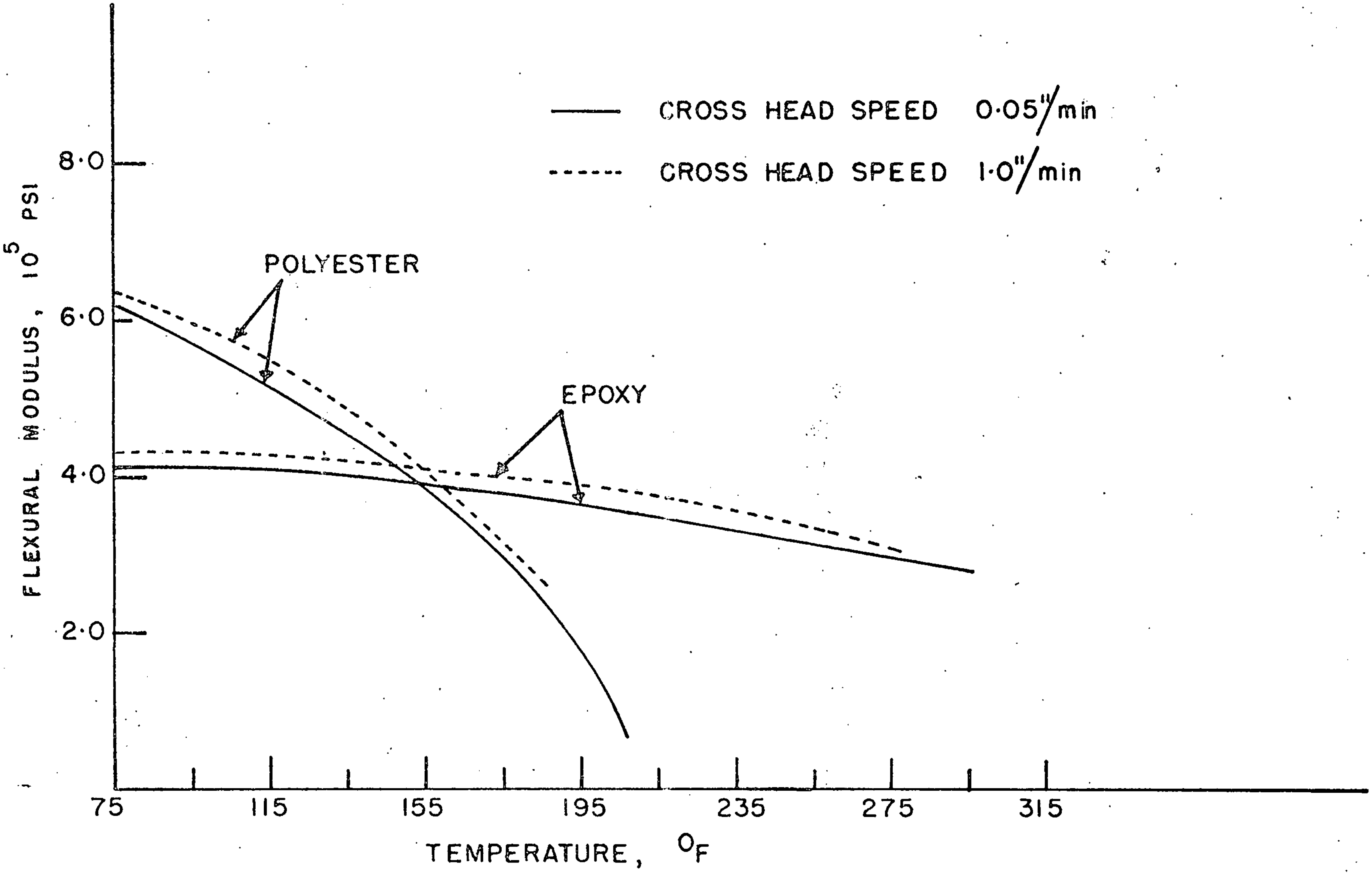


FIGURE 5: VARIATION OF FLEXURAL MODULUS WITH TEMPERATURE

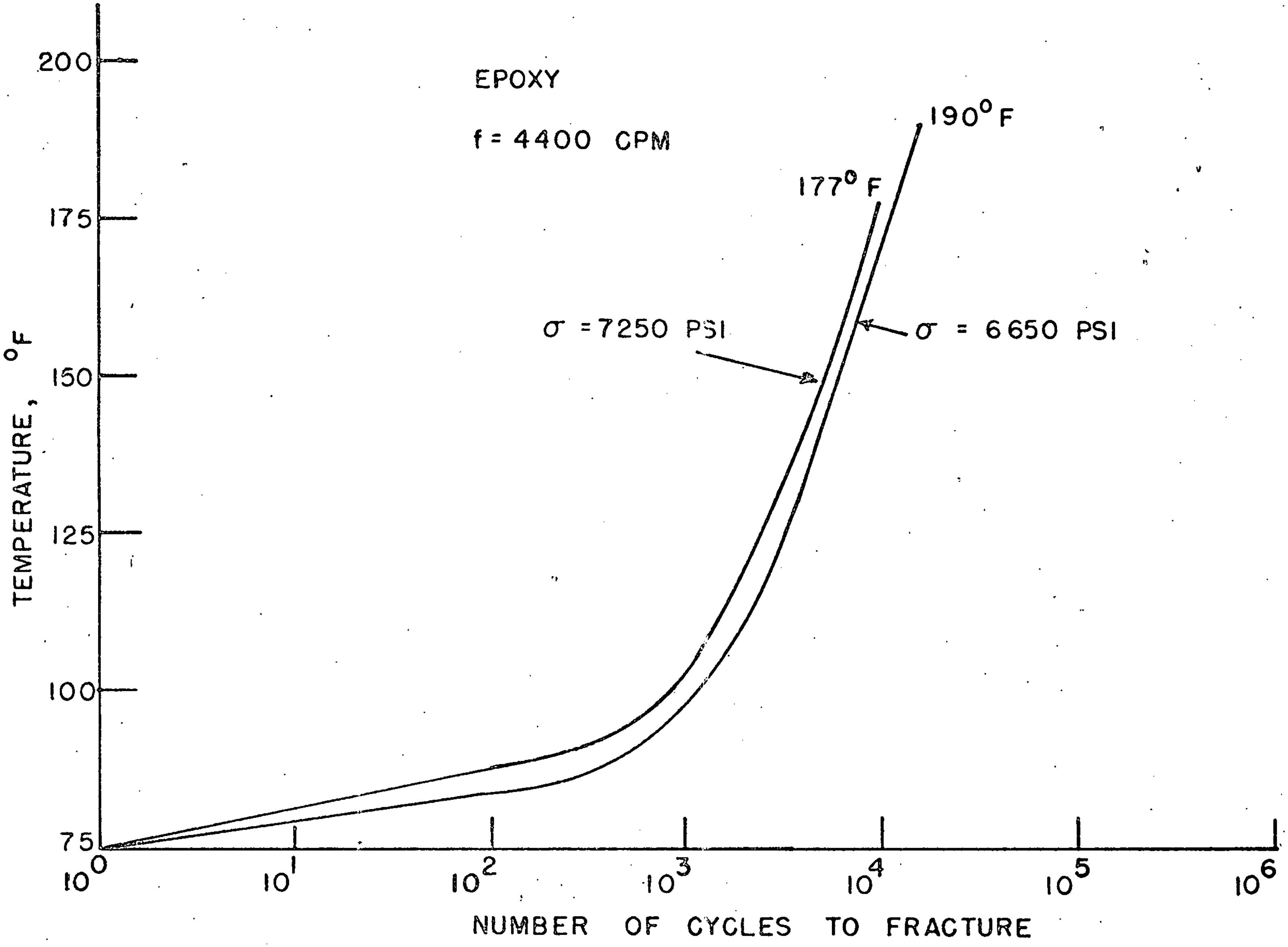


FIGURE 6. TEMPERATURE RISE AS A FUNCTION OF FATIGUE CYCLES

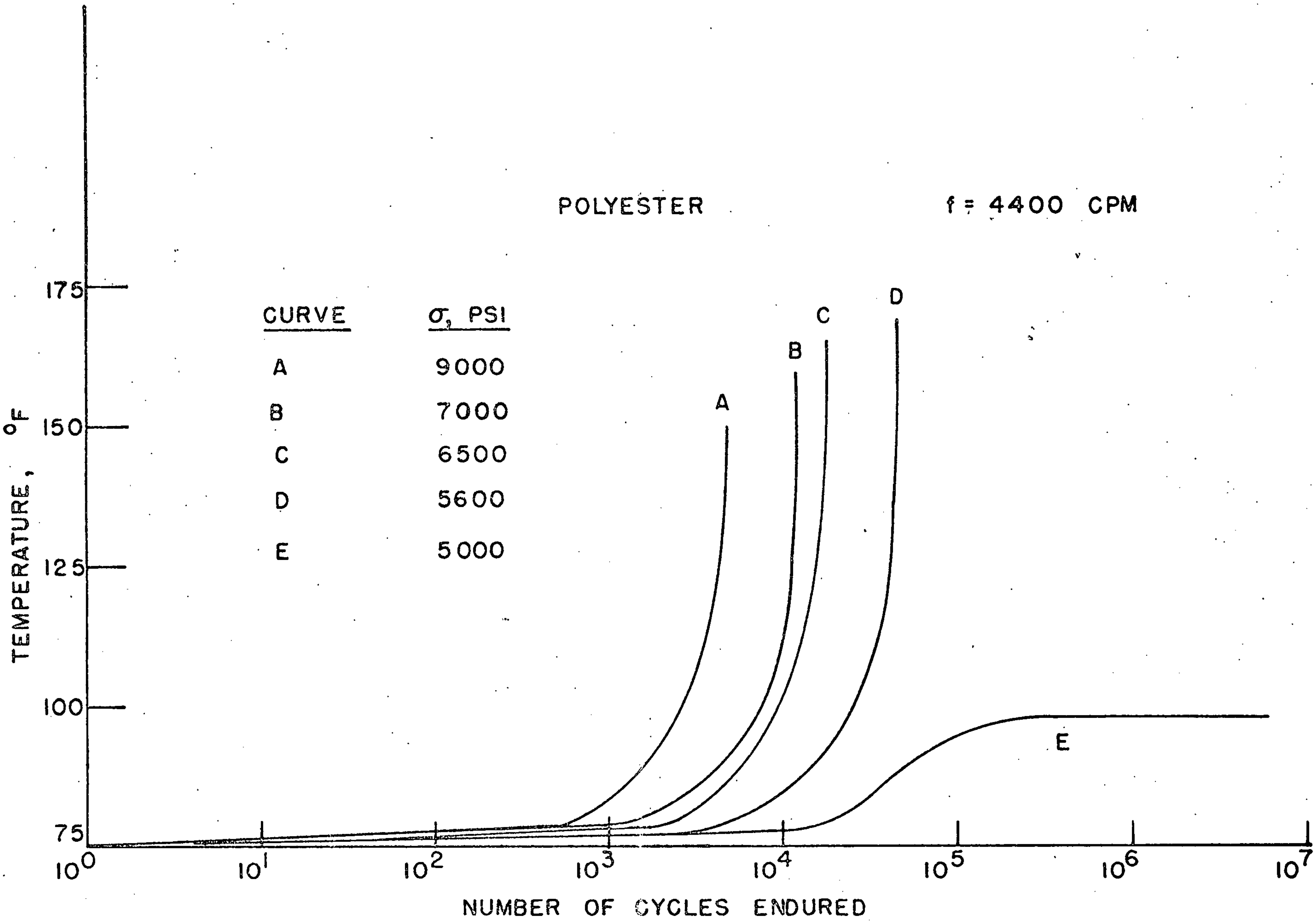


FIGURE 8. TEMPERATURE RISE AS A FUNCTION OF FATIGUE CYCLES

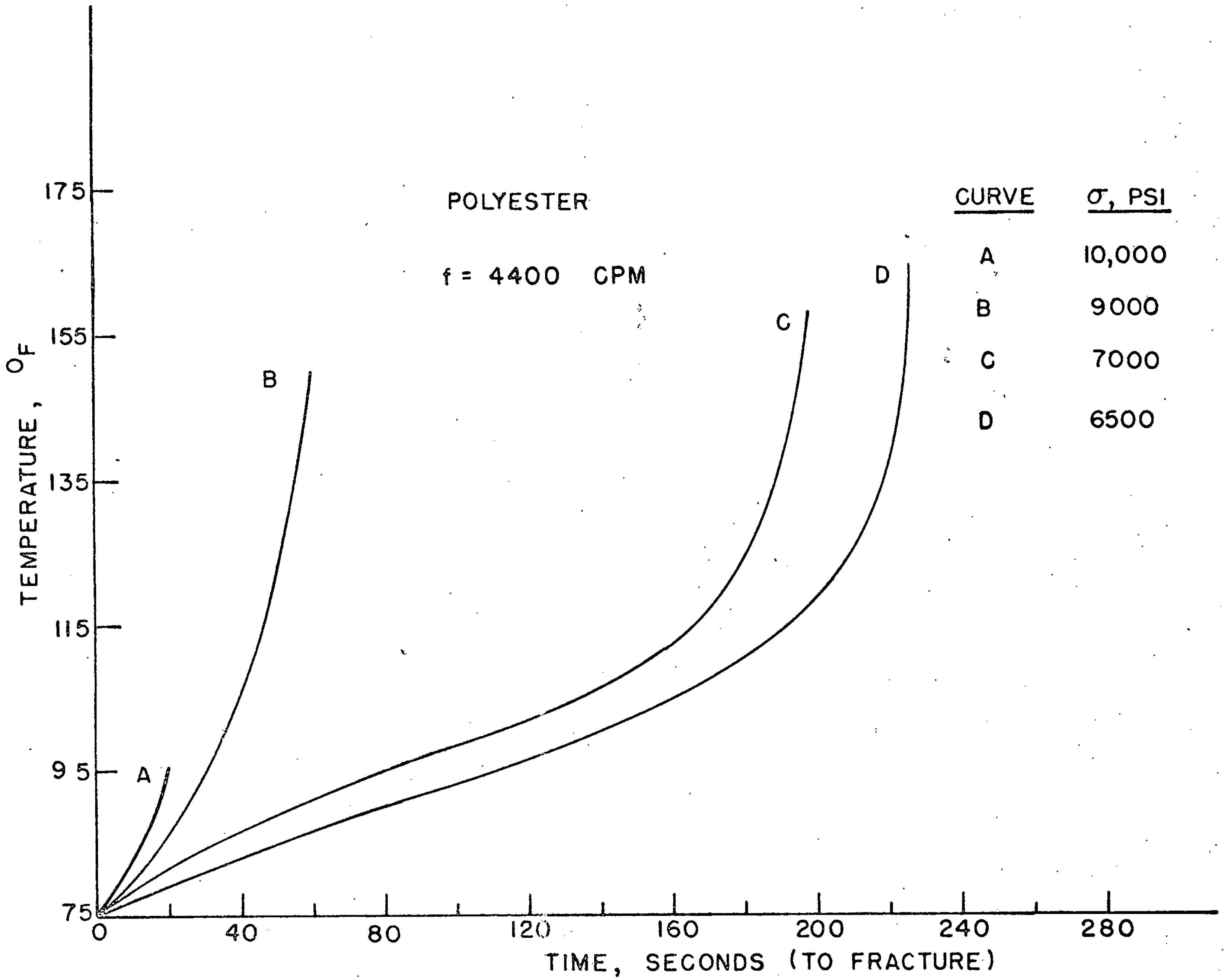


FIGURE 9. TEMPERATURE RISE AS A FUNCTION OF TIME

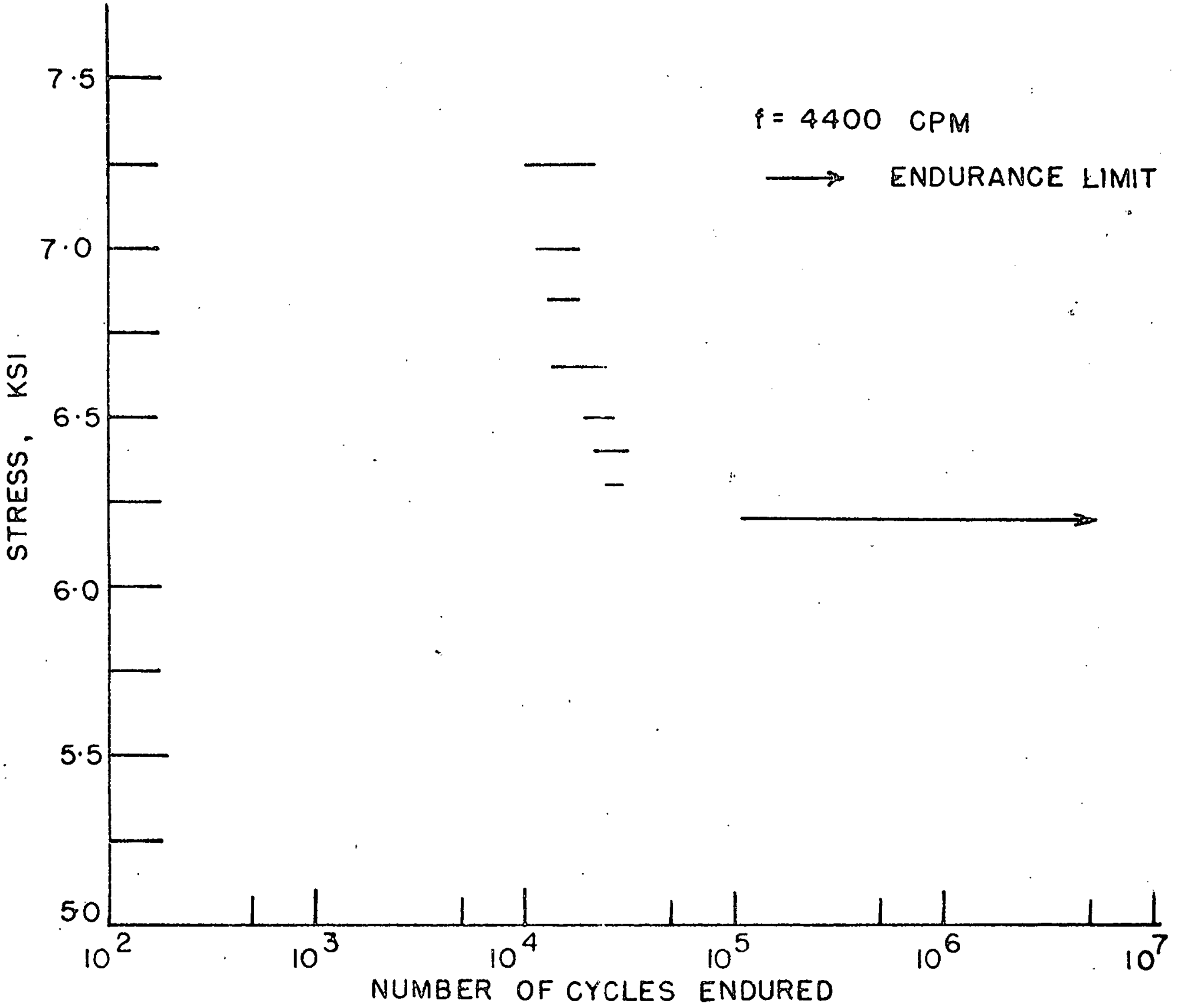


FIGURE 10. S-N CURVE FOR EPOXY RESIN

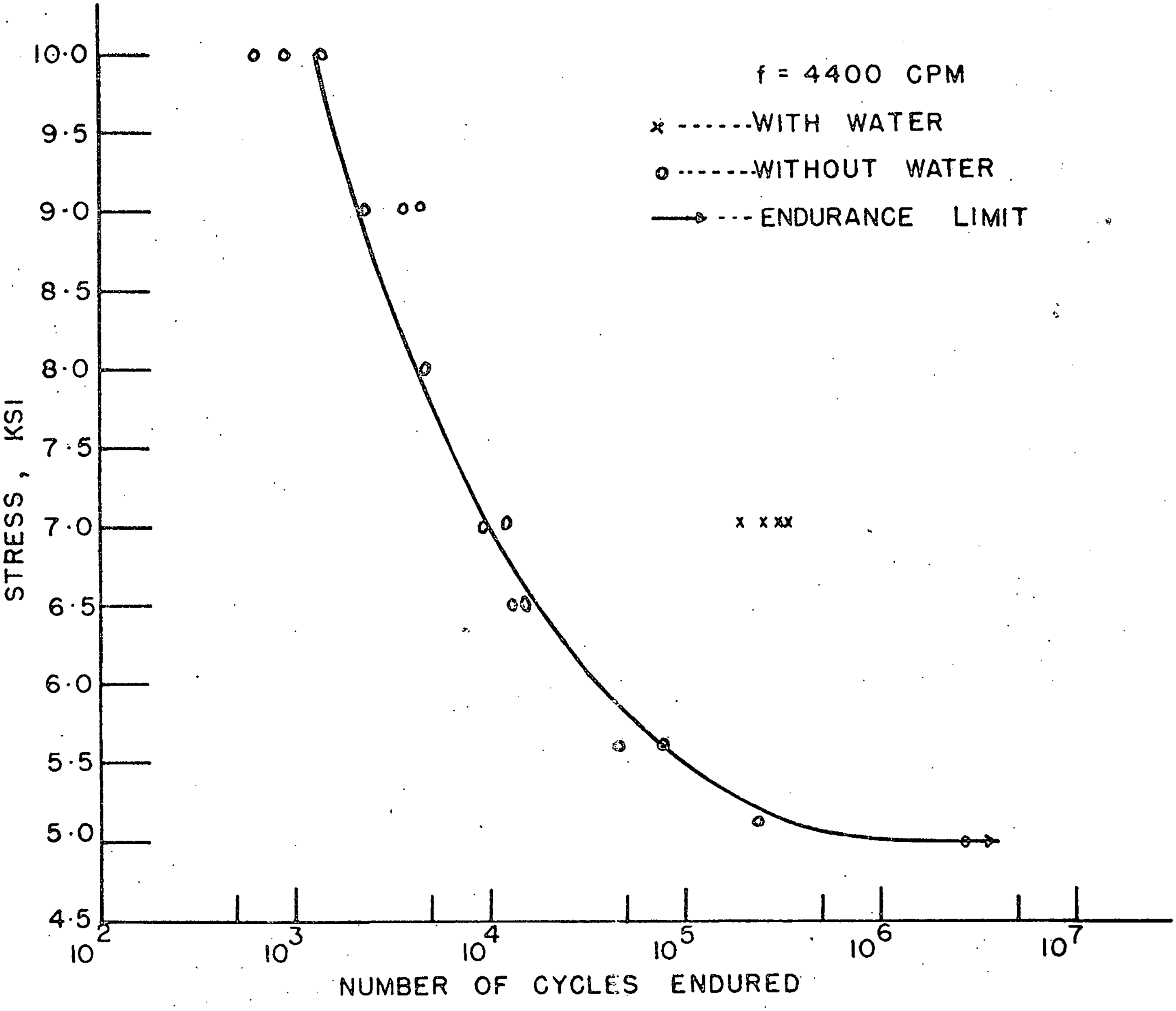


FIGURE 11. S-N CURVE FOR POLYESTER RESIN

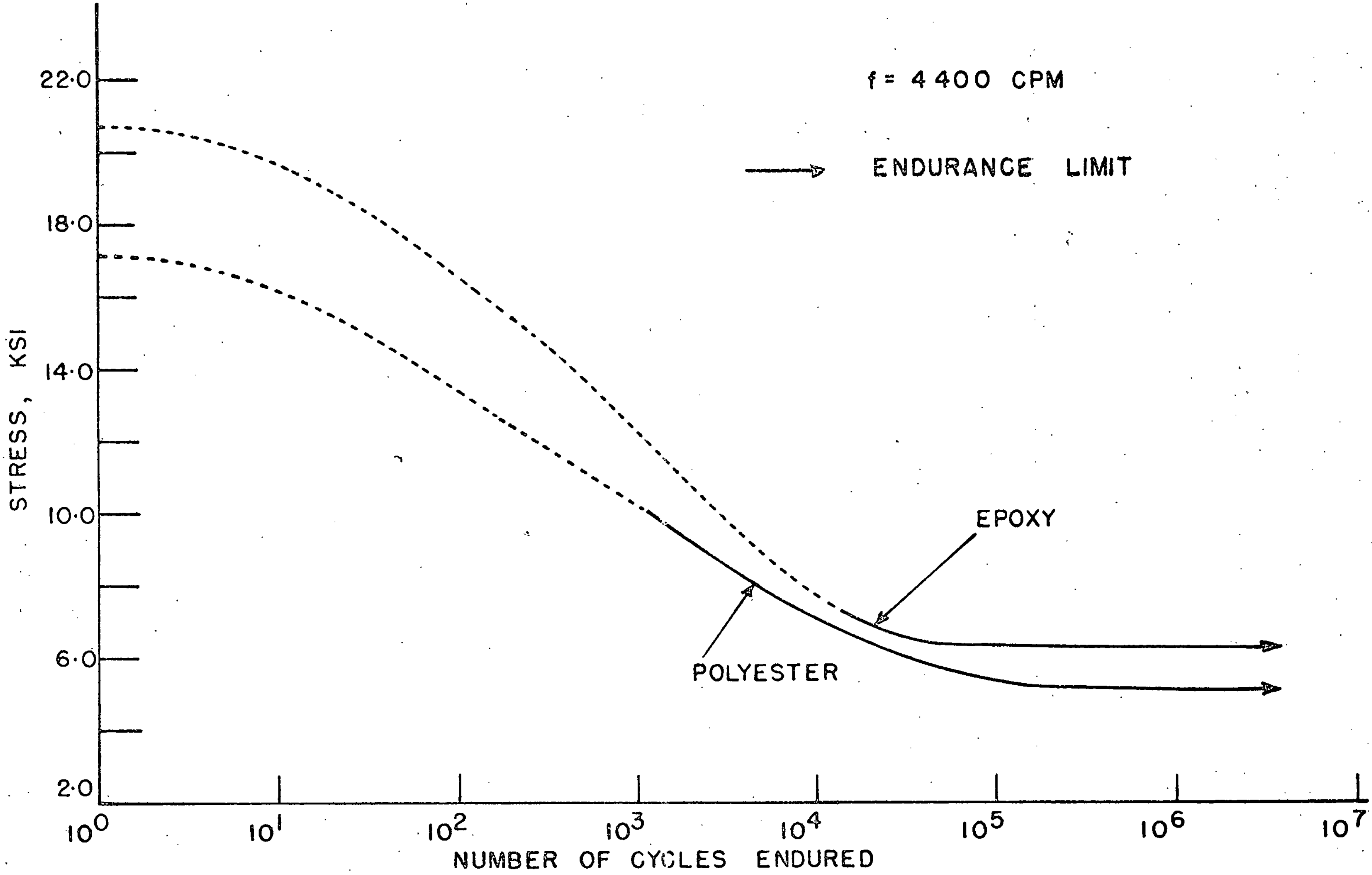


FIGURE 12. S-N CURVES FOR EPOXY AND POLYESTER RESINS

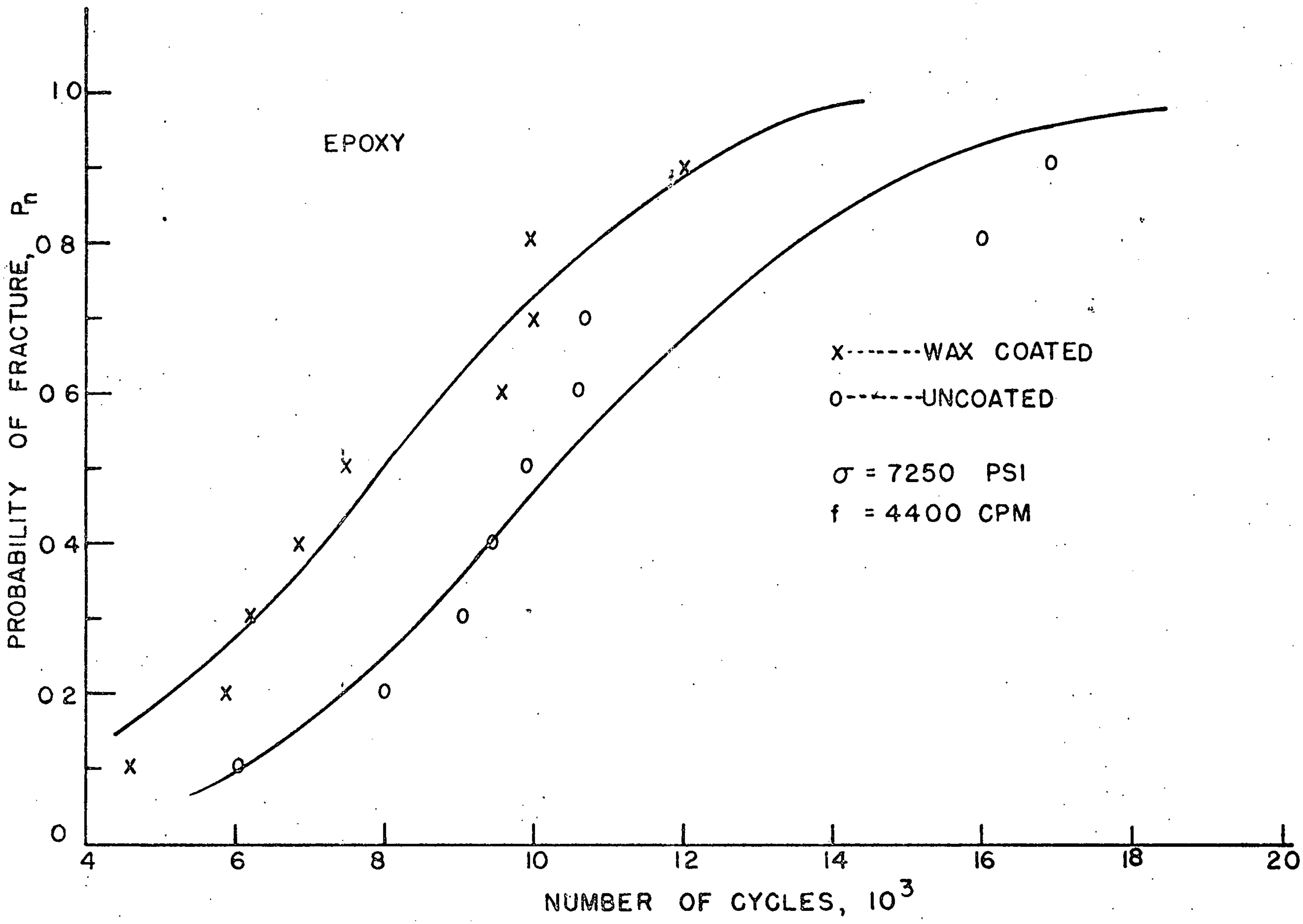


FIGURE 13. EFFECT OF WAX COATING ON PROBABILITY OF FATIGUE FRACTURE

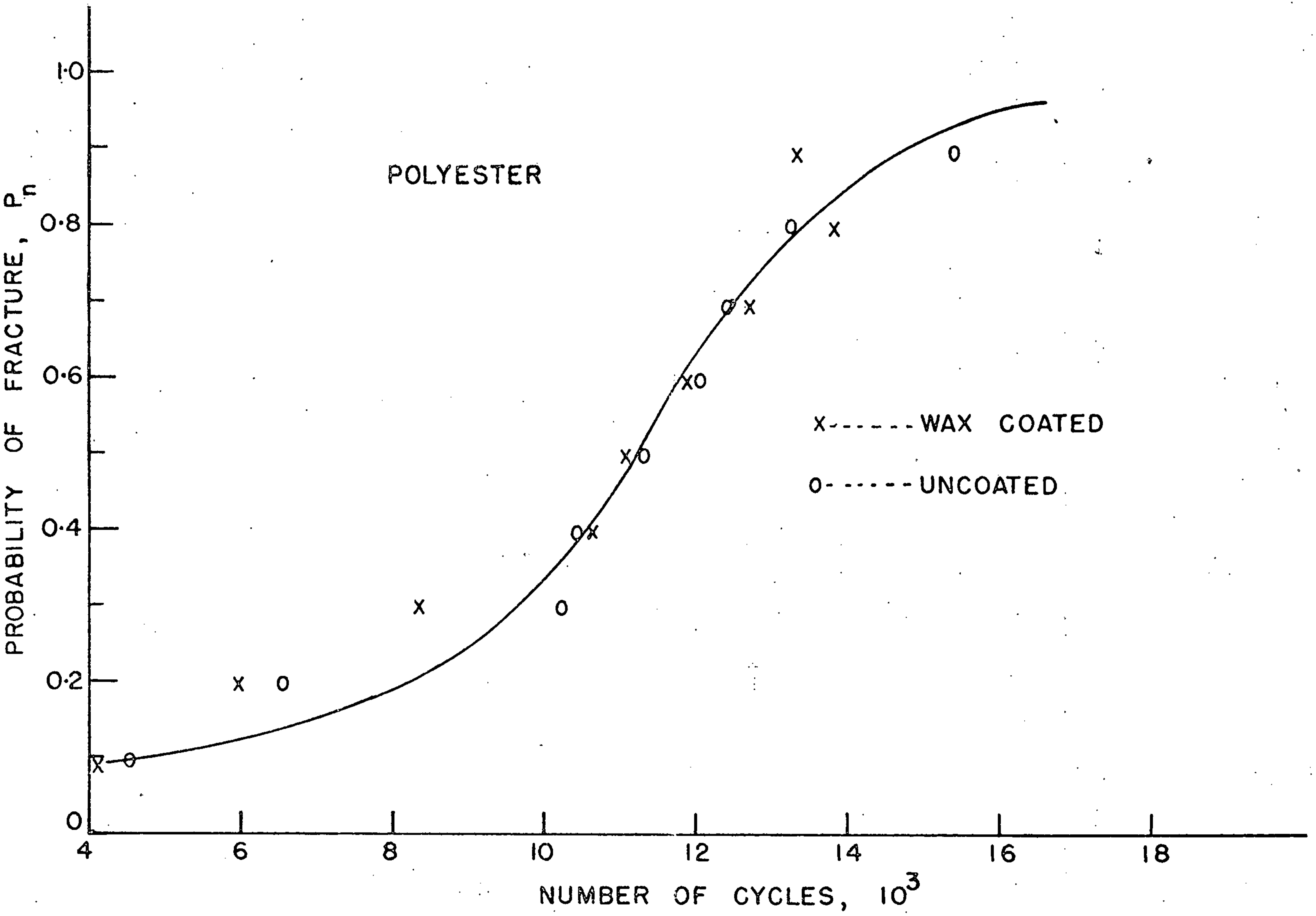
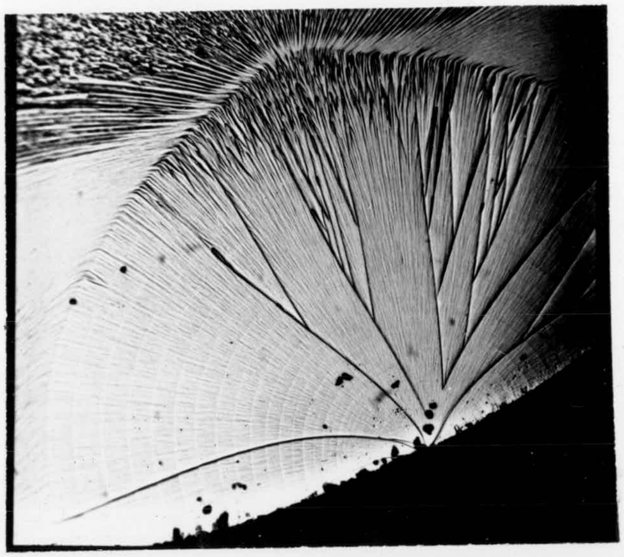


FIGURE 14. EFFECT OF WAX COATING ON PROBABILITY OF FATIGUE FRACTURE

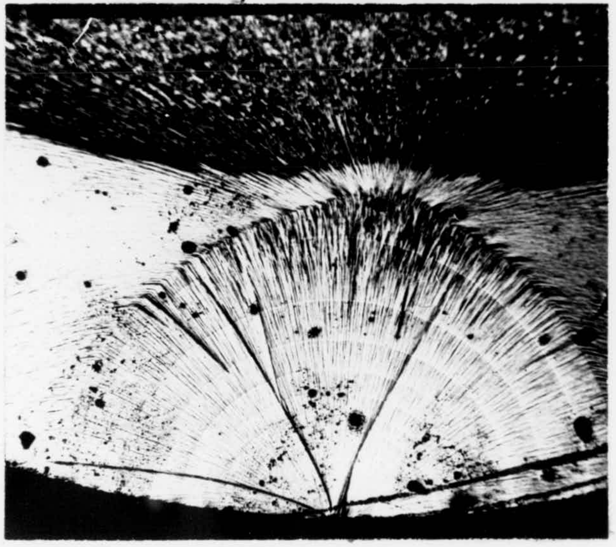
(a) $\sigma = 7250$ PSI, f = 4400 CPM

(b) $\sigma = 6350$ PSI, f = 4400 CPM

FIGURE 15 FRACTURE SURFACES OF EPOXY RESIN ROTATING BEAM FATIGUE SPECIMENS SHOWING FRACTURE ORIGIN (50 X)



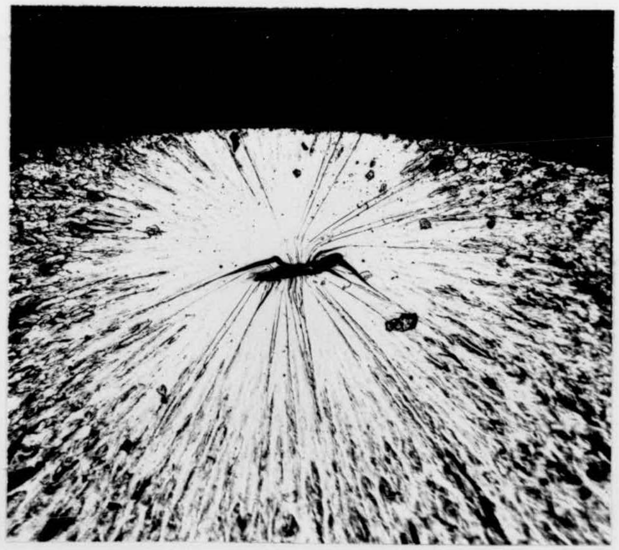
(a) σ = 5600 PSI, f = 4400 CPM



(b)

σ = 6000 PSI, f = 4400 CPM

FIGURE 16 FRACTURE SURFACES OF POLYESTER RESIN
ROTATING BEAM FATIGUE SPECIMENS
SHOWING FRACTURE ORIGIN (50X)



(c) $\sigma = 10{,}000$ PSI, f = 4400 CPM

FIGURE 16 (CONTINUED)