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TRANSVERSE CRACK GROWTH AND FRACTURE OF A THERMOPLASTIC-MATRIX FIBER COMPOSITE AT ROOM AND ELEVATED TEMPERATURES*

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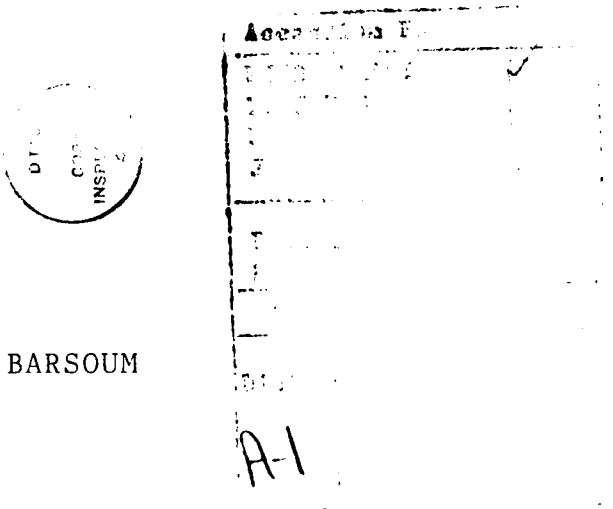
ABSTRACT

A study of transverse crack growth and fracture behavior in a thermoplastic-matrix fiber composite at both room and elevated temperatures is conducted. Owing to the significant ductility of constitutive properties of the thermoplastic composite, a nonlincar fracture mechanics approach is taken in the study. Experiments are conducted on the AS4-fiber/polyamide-matrix composite, using a notched compact tension specimen. The influence of temperature, ranging from room temperature up to glass transition temperature, on transverse crack initiation and growth toughnesses and the final fracture has been studied. It is found that the initiation toughness of transverse crack growth is very sensitive to the temperature change. Contrary to the behavior in the neat thermoplastic J1 resin, an increase in temperature increases the transverse crack initiation toughness. Similar effects of the temperature change on transverse crack growth and fracture are found for the AS4/J1 composite system. At room temperature, low fiber-matrix interface bonding and relative brittleness of the glassy matrix lead to low initiation toughness and crack growth resistance. As the temperature increases, the interface adhesion is improved and high thermoplastic matrix ductility prevalent in the composite, leading to high initiation toughness and stable crack growth resistance. These phenomena are observed and determined both from the experimental and analytical results and from the SEM fractographic study.



1. INTRODUCTION

The use of advanced thermoplastic-matrix fiber composites in high-performance engineering structures, such as supersonic airframes, where elevated temperatures occur, opens up a relatively new field of research [1-4]. Many fundamental problems associated with the thermoplastic-matrix composites are still not well understood, such as effects of temperature, microstructural parameters and processing conditions on thermomechanical properties and failure behavior. This class of advanced materials will not reach their full potentials until these problems are resolved. In this paper, transverse crack growth and fracture of a thermoplastic-matrix fiber composite are studied at both room and elevated temperatures.

The study of elevated-temperature crack growth and fracture behavior of thermoplastic-matrix fiber composites is recognized to involve numerous difficulties, both in experiment and in theory. Previous fracture experiments performed on this type of materials at room and elevated temperatures reveal various complications [5-7]. These involve, for example, the inherently local nonlinear deformation and the associated large geometric change, especially at elevated temperatures, resulting from the intrinsic ductile nature and the well-known time-temperature dependence of the matrix phase [6,8]. Also, problems involving microscopic heterogeneity and material anisotropy of reinforced polymer composites due to the presence of strong fibers lead to additional complexities. Furthermore, thermoplastic-matrix composites are strongly affected by processing conditions, matrix morphology, fiber-matrix interphase microstructure, and multiple fracture modes caused by fiber orientation and lamination [9-11]. These complexities make the problem very difficult to resolve not only from an experimental perspective but also from a rigorous theoretical point of view.

Over the last several years, a significant amount of effort has been made on the study of interlaminar fracture toughnesses of various thermoplastic-matrix composites at room temperature, for example, Refs. [5, 12-14]. To the authors' knowledge, very little information is available in the literature on the subject of elevated-temperature crack growth and fracture of advanced thermoplastic-matrix composites. An attempt is made in this research to provide the much needed information in these subject areas. The objectives of this paper are to: (1) introduce a nonlinear composite fracture mechanics approach for studying the initiation and growth of a transverse crack in a thermoplastic-matrix composite, (2) determine the initiation toughness and crack growth resistance of the composite system, (3) study the effect of elevated temperature on the transverse crack growth and fracture behavior in the semicrystalline thermoplastic composite, and (4) identify fundamental characteristics of the crack growth and fracture in the composite at different temperatures.

In the next section, an experimental program is presented. Information on the thermoplastic-matrix composite system used and the nonlinear fracture experiment procedure conducted in the study are given. A theoretical basis of the nonlinear fracture mechanics approach is briefly introduced in Sec. 3 for the transverse crack growth and fracture study. Results obtained from the experiment and the analysis are given in Sec. 4. Discussion on fundamental characteristics and mechanics of transverse crack growth and fracture in the thermoplastic-matrix composite at several temperature levels is presented. Important conclusions derived from the study are given in Sec. 5.

2. TRANSVERSE CRACK GROWTH AND FRACTURE EXPERIMENT FOR AS4/J1 COMPOSITE AT ELEVATED TEMPERATURES

2.1. Material and Specimen Preparation

The material used in the experiment was provided by Du Pont. The thermoplastic composite is a continuous graphite-fiber reinforced polyamide-matrix composite, referred to as the AS4/J1 composite. The composite was prepared in a molded plate-form with a unidirectional layup made by a melt-impregnation process. A matched-mold molding process was employed at 300^{o} C under 1200 psi pressure in a 6x6 inch steel mold. After solidification, the mold was cooled down to room temperature under pressure. The constitutive elastic properties of the AS4/J1 composite at various temperatures are presented in Table 1 and the complete transverse stress-strain behavior of the composite is given Fig. 1.

The commonly used compact tension specimen was selected for studying the transverse crack growth and fracture in the thermoplastic-matrix composite. The specimens were machined to the dimensions specified in Fig. 2. To obtain an initially sharp crack, 0.5 inch long notch was first machined with a low-speed diamond-blade saw, then sharpened using a razor blade forced into the material in a vise until a desired aspect ratio a/W was obtained. The specimens were cut in such a way that the fibers were oriented perpendicular to the loading direction. The initiation toughness of crack growth was determined for the in-plane transverse direction (or in the 2-direction), according to the coordinate system shown in Fig. 3. To facilitate the crack extension observation and measurement, all specimen surfaces were coated with white correction fluid before the notch was sharpened. This is illustrated in Fig. 4 where both blade saw and razor blade notches can be observed. To insure the clip-gage standing firmly, two knife edges were glued exactly along the load axis. This feature had two advantages: (i) to record the crack

mouth opening displacement directly on the load-line displacement axis, and (ii) to eliminate the problem of the moving rotating point location on the uncracked ligament as the crack initiated and propagated.

2.2 Experimental Procedure

All experiments were performed on an MTS servohydraulic test frame. Software was developed to insure the control of the system and the data acquisition between the MTS control unit and an IBM microcomputer. The temperatures used in the experiments cover the range between the ambient temperature up to the glass transition temperature of the (J1) neat resin. Prior to each test, the environment chamber was set to a selected temperature for half a day to allow temperature stabilization of the equipment, particularly the load cell.

The technique used to determine the crack initiation and growth toughnesses of the AS4/J1 composite was carried out for the notched compact tension specimen. The analytical method is suitably developed and allows proper data reduction. A view of the experimental setup is presented in Fig. 5. Note the clip-gage used to measure the crack mouth opening displacement. The clip-gage slots were located exactly on the loading axis. Note also the coating used to facilitate observations of the crack tip and to follow the crack growth path. Before each test, the initial crack length was measured with a travelling tele-microscope.

The exact location of the crack tip was sometimes difficult to determine due to the misalignment of fibers, which prevented the operator from differentiating crack opening from crack extension. The experimental procedure followed a similar scheme as the one used for studying the (J1) neat resin [15]. The load-point displacement rate was set to 10^{-4} in/s which allowed proper observation of the crack offset. Three specimens were tested at each temperature. After initiation, the crack growth was followed by using a travelling tele-microscope through the window of the environment chamber. The location of the crack tip was noted continuously at regular intervals along with the corresponding load and crack-

mouth opening displacement. At this stage of the experimental, the load P, the load-line displacement δ, and the corresponding crack extension Δa were recorded for further analysis based a nonlinear fracture mechanics approach.

3. ANALYSIS OF TRANSVERSE CRACK GROWTH AND FRACTURE

3.1 Nonlinear Fracture Mechanics Considerations

In view of the significant material nonlinearity exhibited by the thermoplastic-matrix composite system, a nonlinear fracture mechanical approach is taken to study quantitatively various stages of the fracture process in the composite. Within the context of mechanics of composite fracture, two issues have been of significant interest in this study: governing parameters for characterizing the transverse crack initiation and growth toughnesses and fracture resistance, and mechanisms and micromechanics of local crack growth and material damage. Owing to the complex heterogeneous fiber-matrix microstructure and the inherent nonlinearity of the thermoplastic resin, the solution for accurate micromechanical modeling of the local crack-tip field with detailed fiber/matrix interaction, coupled with local material damage and inelastic constituent properties of each phase, is not yet available. The current effort is focused on the determination of transverse crack initiation and growth resistance of the composite and the characteristics associated with the different stages in the composite failure process. This has been made possible by the introduction of the well-known conservation integral, J [16], as a suitable parameter in the study of the nonlinear anisotropic composite fracture problem.

Consider the thermoplastic-matrix composite as a homogeneous anisotropic nonlinear solid. The conservation integral J introduced in [17,18] may be used here in the analysis,

$$J = \int_{\Gamma} \left(W n_1 - \sigma_{ij} n_j \frac{du_i}{dx_1} \right) ds \qquad (1)$$

where Γ is a contour encircling the crack tip counterclockwise; W is the strain energy density function, n_j are outward normals to Γ, and σ_{ij} and u_j are stress and displacement components, respectively. In the context of the nonlinear fracture mechanics, the initiation of crack growth is assumed when the applied J reaches the material initiation toughness, J_{Ic},

$$J(a,P) = J_{IC} \tag{2}$$

where a and P are the crack length and the applied nominal load respectively, provided that the crack-tip process zone is small and embedded in the J-dominant field.

During the crack growth in a nonlinear solid, the local material resistance generally changes with crack extension. The J vs. Δa curve, referred to as the resistance curve, is commonly used to describe the nature of the stable growth of the crack in the material during fracture. Paris [19,21] suggests that the slope of the stable crack growth curve may be used to represent the crack growth driving force. For a crack growth problem in the thermoplastic composite, a similar expression may be used,

$$T = \beta \frac{dJ}{da} \tag{3}$$

where T is called the tearing modulus and β is a function of elastic moduli C_{ij} and yield stresses σ_{oi} of the composite. We note that during crack growth, the crack-tip field is more complex than that in a stationary crack case. Nonproportional loading, history dependence of material nonlinear constitutive equations and local microscopic cracking make the tip region of a growing crack different significantly from the one associated with a stationary crack. Since general anisotropic nonlinear constitutive equations for a thermoplastic-matrix composite are not yet fully available, detailed expressions for incremental crack-tip stress

and strain fields can not be written in the same form as those proposed for isotropic nonlinear materials.[19,20].

3.2 Determination of Crack Initiation and Growth Resistance

Based on an energy approach, it has been shown [22] that for a deeply notched specimen if the inelastic deformation is restricted to the uncracked ligament region, b=W-a, then the J integral can be approximated by

$$J \simeq \frac{2}{Bb}\int_{o}^{\delta_c} P\, dv \tag{4}$$

where δ_C is the crack opening displacement; B is the specimen thickness, and P is the applied load. An alternative form for Eq. (4) is given [23,24] as

$$J = \frac{\eta U_c}{Bb} F(a/w, C_{ij}) \tag{5}$$

where U_C is the area under the P-δ curve owing to the introduction of the crack. The η and $F(a/w, C_{ij})$ are functions of specimen and crack geometry and material properties, which will be discussed in this section. Thus the J may be calculated directly from the initial ligament length b and the load-displacement diagram for a deeply notched compact-tension specimen.

The factor η in Eqs. (5) is a calibration factor, depending on both the specimen geometry and the nature of the deformation occurring [23,25], and can be defined as

$$\eta = \frac{(W-a)}{\lambda}\frac{d\lambda}{da} \tag{6}$$

where λ is the material transverse elastic compliance. The numerical analysis in [23] has shown that for a deeply notched specimen subjected to pure bending, this factor converges

to 2 as long as the aspect ratio a/W remains greater than 0.5. For the composite compact-tension specimen currently used, the $F(a/W, C_{ij})$ is expressed as

$$F(a/w,C_{ij}) = \frac{1+\alpha}{1+\alpha} f(C_{ij}) \qquad (7)$$

with

$$\alpha = 2\left[\left(\frac{a}{b}\right)^2 + \frac{1}{2}\right]^{\frac{1}{2}} - 2\left[\frac{a}{b} + \frac{1}{2}\right] \qquad (8)$$

The $f(C_{ij})$ is a dimensionless quantity to account for the anisotropy of the composite material. Current numerical studies for the AS4/J1 thermoplastic composite specimen reveal that $f(C_{ij})$ has a value approximately 1.22.

4. RESULTS AND DISCUSSION

As indicated in Sec. 2, the AS4/J1 thermoplastic-matrix composite is noted to exhibit significant nonlinearity in its constitutive properties. The nonlinearity increases with temperature as shown in Fig. 1. This situation is expected since the thermoplastic matrix, i.e., the neat J1 resin, possesses an inherent ductility and experiences a similar degree of sensitivity to the change of temperature, as reported in [8,15]. However, at a given temperature, the failure strain of transverse deformation in the thermoplastic composite is noted to be approximately one order of magnitude lower than that of the neat resin owing to the complex nature of the composite morphology and microstructure and the microscopically constrained matrix flow in the heterogeneous system. The significant material nonlinearity of the composite warrants the proper use of the nonlinear fracture mechanics approach to the current transverse crack growth and fracture problem. We note that the requirements for a valid J-controlled crack growth in the AS4/J1 thermoplastic composite have been checked at all temperatures studied, and all the results obtained generally satisfy the constraint conditions and the applicability of the J-integral to the initiation and stable crack growth in the composite fracture. The resistance curves for transverse crack initiation and growth in the AS4/J1 composite have been determined for various cases. The effect of temperature on crack initiation toughness and crack growth resistance has been studied and compared with those of the neat resin material. Scanning electron microscopy has also been carried out to examine the failure characteristics in the initiation and propogation regions of the fracture surface.

4.1 Effect of Temperature on Crack Initiation Toughness J_C

The effect of temperature on the critical J value for transverse crack initiation is shown in Fig. 6 for the AS4/J1 thermoplastic composite. For comparison, the values of J_C

for a compression-molded J1 neat resin at the same temperature levels are shown. Below the glass transition temperature T_g = 145° C, increasing T leads to an appreciable increase in the J_c value of the AS4/J1 composite. Above T_g, a very high level of crack initiation toughness of the material is obtained. We remark that at T_g, the absolute value of the high J_c may not be meaningful as the viscoplastic flow of the matrix becomes dominant. The increase in the J_c value with temperature is thought to be associated with the improved fiber/matrix interface adhesion and increased local matrix ductility in the composite. It is interesting to note that, opposite to what is observed in the AS4/J1 composite, the value of J_c of the neat thermoplastic J1 resin decreases significantly with increasing temperature.

Comparing the critical J value of the AS4/J1 material with that of the compression molded J1 neat resin, one observes a large difference at 25° C. The J_c value of the AS4/J1 composite is about 3 psi-in. compared to 13 psi-in. for the compression-molded J1 resin. The decrease in the crack initiation toughness is probably due to a weak fiber-matrix interface bond and the embrittlement of the glassy matrix, induced by the presence of fibers, at room temperature. (The SEM micrograph Fig. 9(a) of the crack initiation region at 25° C show the bare fiber surface, suggesting poor bonding of matrix to fiber. Also, the matrix in the composite does not seem to exhibit much ductility compared with the neat resin behavior at the same temperature.) At high temperatures, the critical J values of the matrix and the composite become close, because the strength of the interface adhesion is improved and the matrix ductility in the composite increases, which will be shown later in the SEM fracture micrograph, Figs. 9(b) - 9(d). The fracture initiation of the transverse crack in the composite is apparently controlled mainly by the matrix resin; thus, results of the initiation toughness of the composite are close to that of the thermoplastic neat resin.

4.2 Resistance Curves for Transverse Crack Growth at Different Temperatures

The J vs. Δa resistance curves for transverse crack growth in the AS4/J1 thermoplastic composite have been obtained at different temperatures, as shown in Figs.

7(a)-7(d). At room temperature, brittle fracture with unstable crack growth is observed. The brittle and unstable nature of the fracture, resulted from the local microstructure and defects, contributes to scatter in the measurement and calculation of the tearing modulus. Similar scattering of the stable crack growth resistance curves is also observed at high temperatures, caused by extensive fiber bridging due to local fiber misalignment and the discrete, stepwise crack extension in a discontinuous mode. In fact, the scatter range is found to be even larger than that at room temperature (Fig. 8). For instance, the transverse crack growth at 120° C is observed to be in a stable manner until the tip is sharpened significantly, then at this point of re-initiation, it would surge forward unstably. The matrix in front of the crack tip may increase in temperature by a small amount during this surge, and thus, increasing the ductility of the matrix, which will blunt the crack tip and stop the crack growth. This process is then repeated.

While a significant amount of scatter of the value of dJ/da is observed in the J vs. Δa resistance diagram at each temperature studied, the effect of high temperature on the crack growth and fracture of the thermoplastic-matrix composite is clearly seen from the results. Increasing the temperature increases appreciably the dJ/da value, i.e., the transverse crack-growth resistance of the composite, due to the microscopic matrix ductility increase in the composite, which will be discussed further in the next section. Above the glass transition temperature T_g, the J-integral approach needs to be modified to account for the significant viscoplastic matrix deformation. We note that the dJ/da values obtained at all temperatures studied in this report satisfy the requirements for a valid J-controlled crack growth and fracture, i.e., $dJ/da > 10$ and $R > 10\ D$, for the thermoplastic composite system currently used.

4.3 Fractographic Study

Scanning electron microscopic (SEM) studies are conducted on the fracture surfaces of the AS4/J1 thermoplastic-matrix composite at different temperatures to identify unique

characteristics of the crack growth and fracture in the material system and to assist in interpretation of the nonlinear fracture mechanics results from the experiments. As anticipated from the J_C vs. T and J vs. Δa results presented in the previous sections, the temperature has a significant effect on transverse crack growth and fracture in the composite. The fracture surface micrograph taken at room temperature, Fig. 9(a), shows a significant amount of clean fiber surfaces as well as a small number of fibers with the matrix still being attached to. The poor interface bonding may contribute to the low transverse crack initiation toughness J_C and crack growth resistance dJ/da in the AS4/J1 composite. The increase in temperature apparently improves the adhesion of the matrix onto the fibers, as shown by a decreasing amount of clean fibers at the fracture surface (Figs. 9(b) - 9(d)). The corresponding increase in values of the J_C and dJ/da with T are clearly shown in Figs 7(b)-7(d) and Fig 8. At 145^{o} C, the matrix is subjected to large viscoplastic deformation between the fibers, as shown by the large amount of drawn resin fibrils still being attached to the fibers. The viscoplastic deformation of the matrix between the fibers is typical of the matrix-dominated transverse fracture at a high temperature. The very high values of J_C and dJ/da at this temperature clearly demonstrate the significant effect of the viscoplastic flow on the high crack initiation and growth toughnesses of the thermoplastic-matrix composite.

5. CONCLUSIONS

A study on the transverse crack growth and fracture of a thermoplastic-matrix fiber composite at room and elevated temperatures has been conducted. Owing to the significant nonlinearity in the composite constitutive equations, a nonlinear fracture mechanics approach has been taken in the study. Transverse crack initiation and growth experiments have been conducted on a notched AS4/J1 polyamide thermoplastic composite. The initiation toughness and transverse crack growth resistance of the AS4/J1 composite have been determined at several temperatures, ranging from room temperature up to the glass transition temperature. Scanning electron microscopy has also been conducted on the fracture surface of the composite to identify the characteristics of the crack growth and fracture behavior at different temperatures.

Based on the experimental and analytical results obtained from the study, the following conclusions may be drawn:

1. Owing to the significant material nonlinearity, the use of a nonlinear fracture mechanics approach is necessary to study transverse crack growth and fracture behavior in the thermoplastic-matrix fiber composite.
2. The initiation toughness, J_C, for transverse crack growth in the AS4/J1 thermoplastic composite increases with temperature. This phenomenon is opposite to the J_C vs. T results for a neat polyamide thermoplastic resin obtained in an accompanying report. Near and above the glass transition temperature Tg, significant viscoplastic flow and damage occur near the crack tip, and modifications of the J-integral approach are needed.
3. The J vs. Δa resistance curves for transverse crack growth in the AS4/J1 composite are well behaved at all temperatures studied. An appreciable amount of data scatter in the crack growth region is observed and the range of the data scatter increases

with temperature, owing to the fiber bridging phenomenon at the crack tip, caused by local fiber misalignment and discrete, stepwise crack growth in the composite.

4. The transverse crack growth toughness, characterized by da/dJ, increases with temperature in the AS4/J1 composite. This is consistent with the crack growth characteristics of the neat polyamide resin reported in [15], and indicates further the matrix-dominant nature of the transverse crack growth and fracture behavior in the composite at elevated temperatures.

5. From the scanning electron microscopy study, it is clear that at room temperature, the poor fiber/matrix interface bonding and the glassy matrix phase result in both low initiation toughness and low crack growth resistance. As the temperature increases, the improved interface adhesion/bond strength and the significant local matrix plastic flow increase both the initiation toughness and the crack growth resistance in the notched AS4/J1 composite material.

6. ACKNOWLEDGEMENT

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TABLE 1

Mechanical Properties of AS4/J1 Thermoplastic Composite

at Room and Elevated Temperatures

T (°C)	25	100	120	145	170	190
E_{11} (MSI)	17.9	---	17.4	---	---	---
E_{22} (MSI)	0.90	0.80	0.67	0.46	0.34	0.27
E_{33} (MSI)	1.04	0.99	0.92	0.75	0.51	---
G_{12} (MSI)	0.77	0.55	0.52	0.40	0.30	---
ν_{12}	0.313	---	---	---	---	---
ν_{21}	0.014	---	0.013	---	---	---

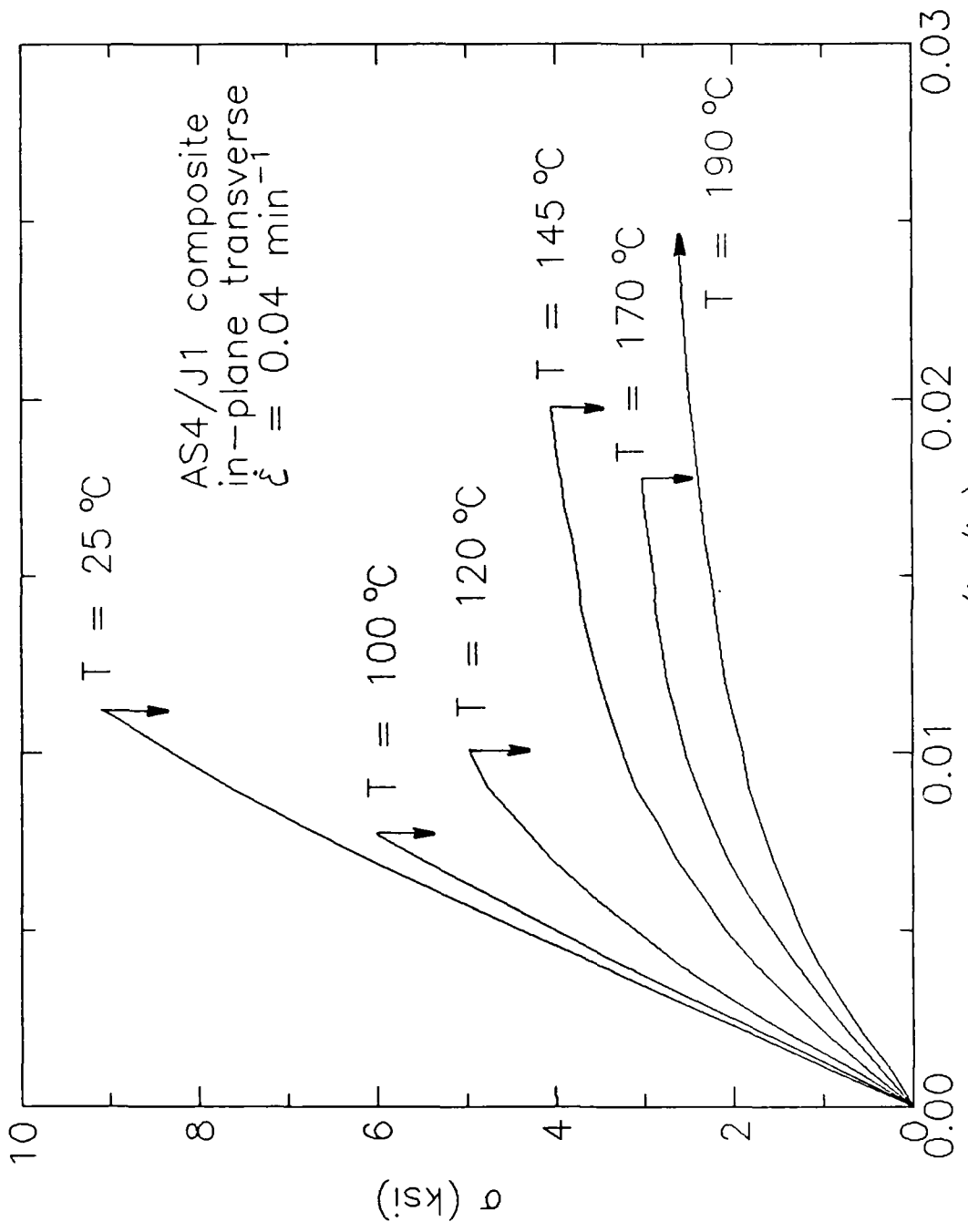

Fig. 1 Effect of Temperature on Transverse Tensile Stress-Strain Behavior of AS4/J1 Thermoplastic Composite.

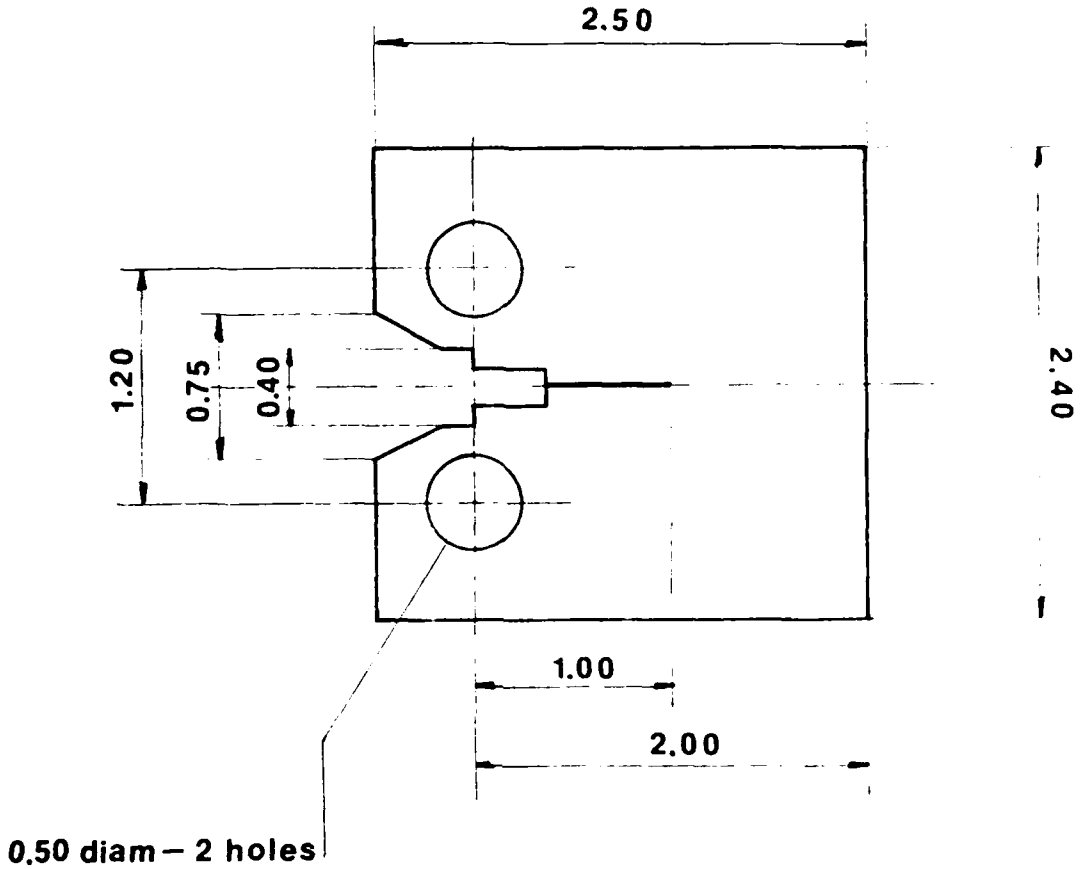

Fig. 2 Specimen Geometry used for Studying Crack Growth and Fracture of AS4/J1 Composite (all dimensions in inches).

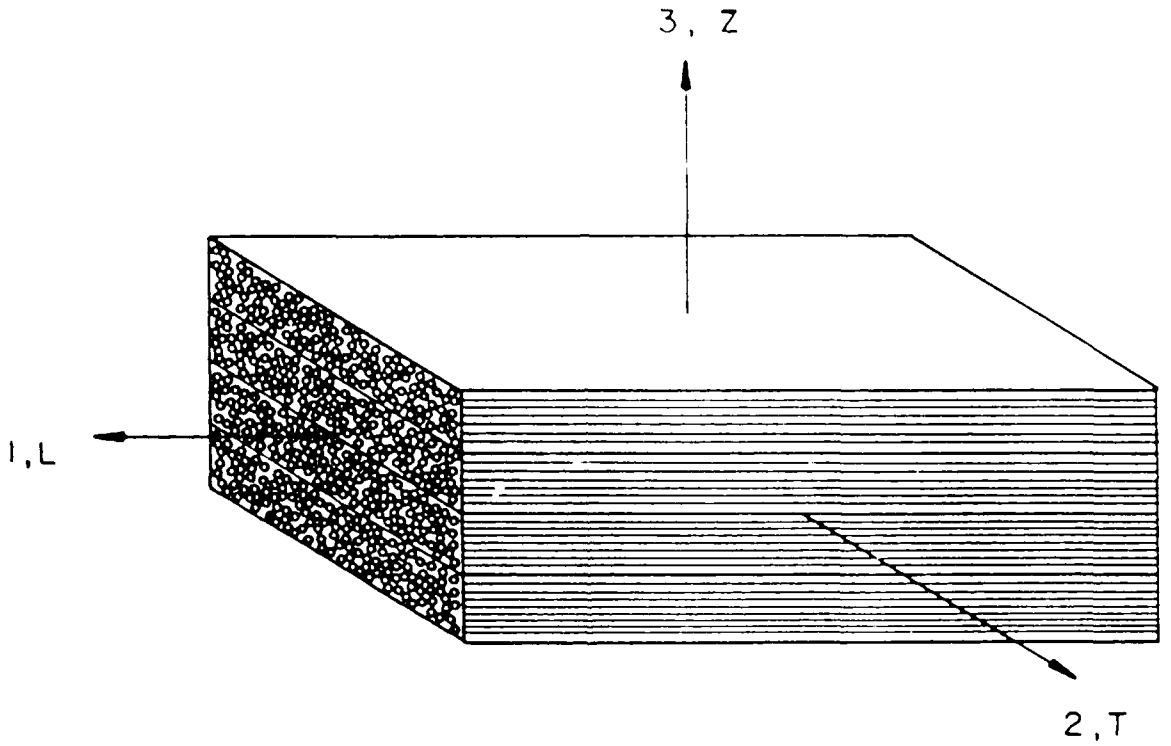

Fig. 3 The Coordinate System and Notations.

Fig. 4 View of the Crack Tip Region in a Compact Tension AS4/J1 Composite Specimen used for Transverse Crack Growth and Fracture Study.

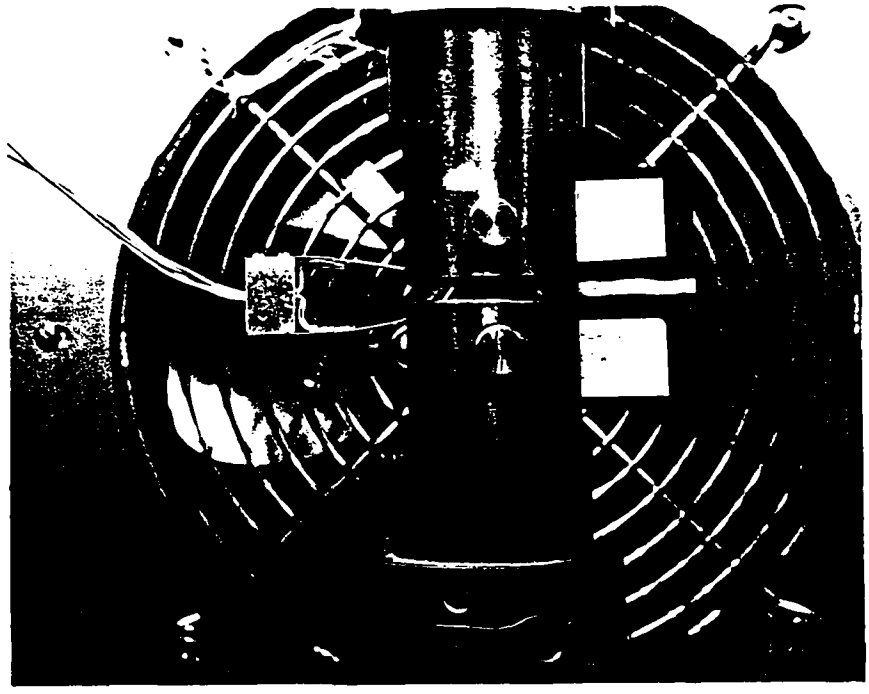

Fig. 5 Experimental Setup for Studying Crack Growth and Fracture of AS4/J1 Composite in an Environmental Chamber.

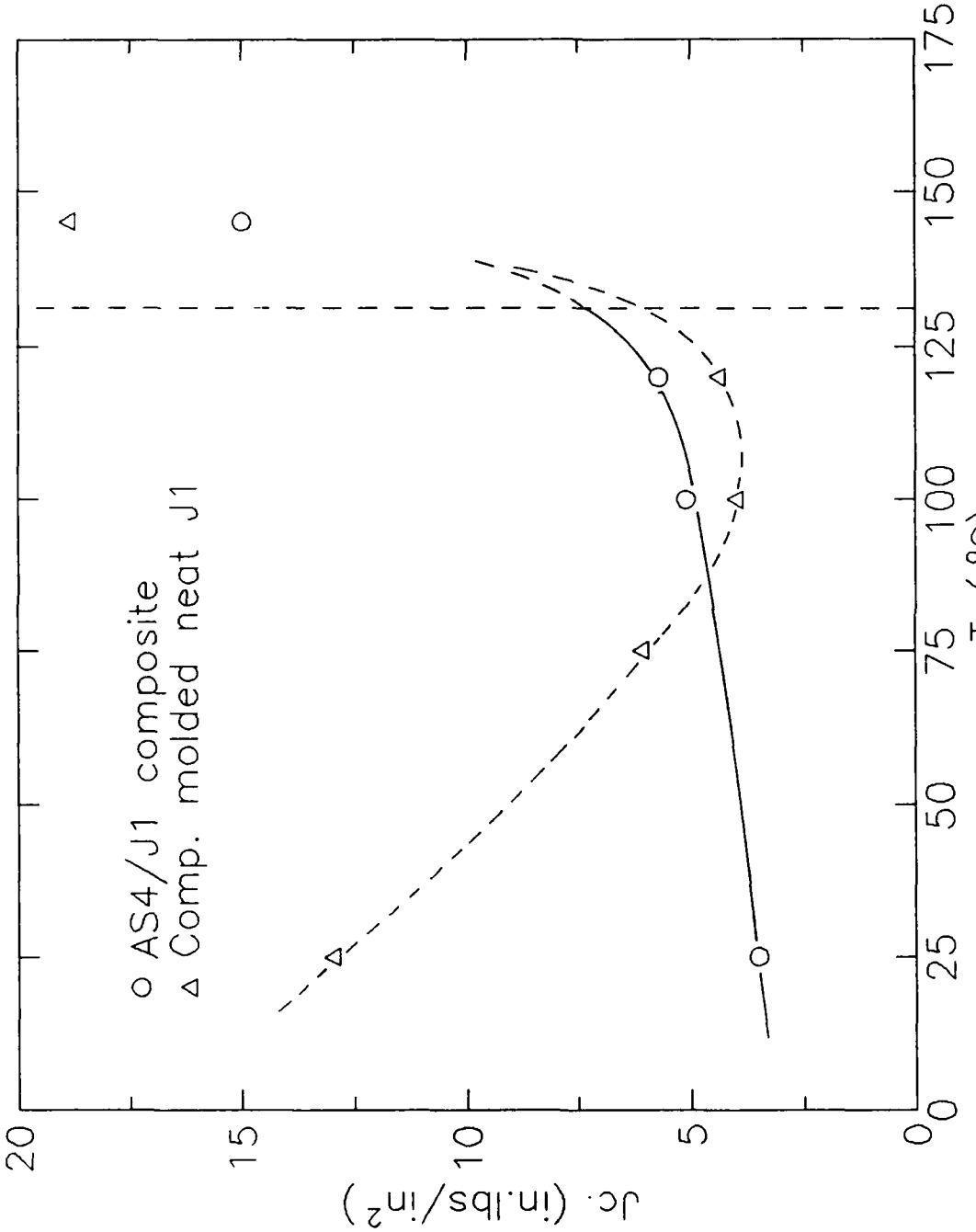

Fig. 6 Transverse Crack Initiation Toughness J_c vs. Temperature T for AS4/J1 Composite.

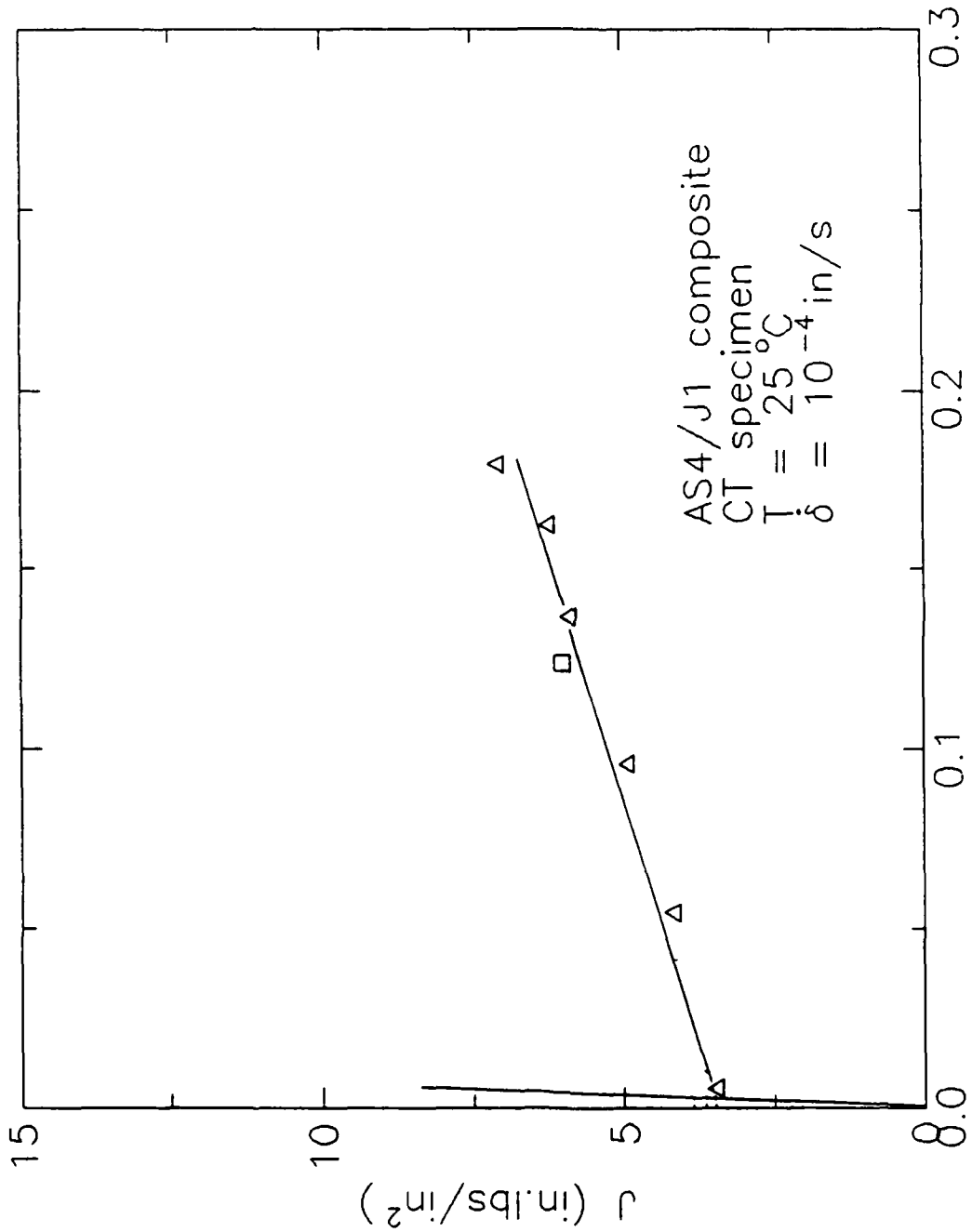

Fig. 7(a) J vs. Δa for AS4/J1 Composite. (T = 25°C)

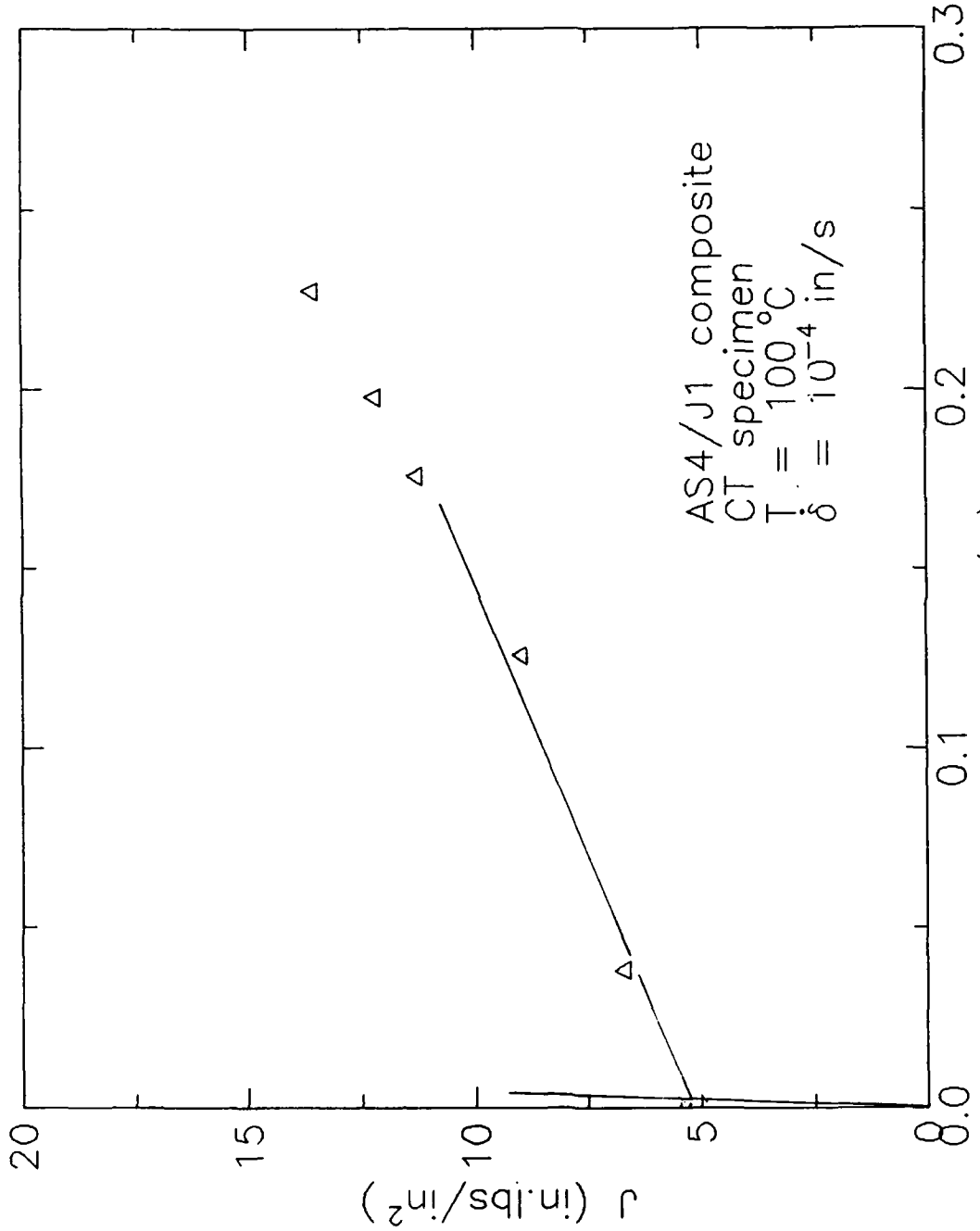

Fig. 7(b) J vs. Δa for AS4/J1 Composite. (T = 100°C)

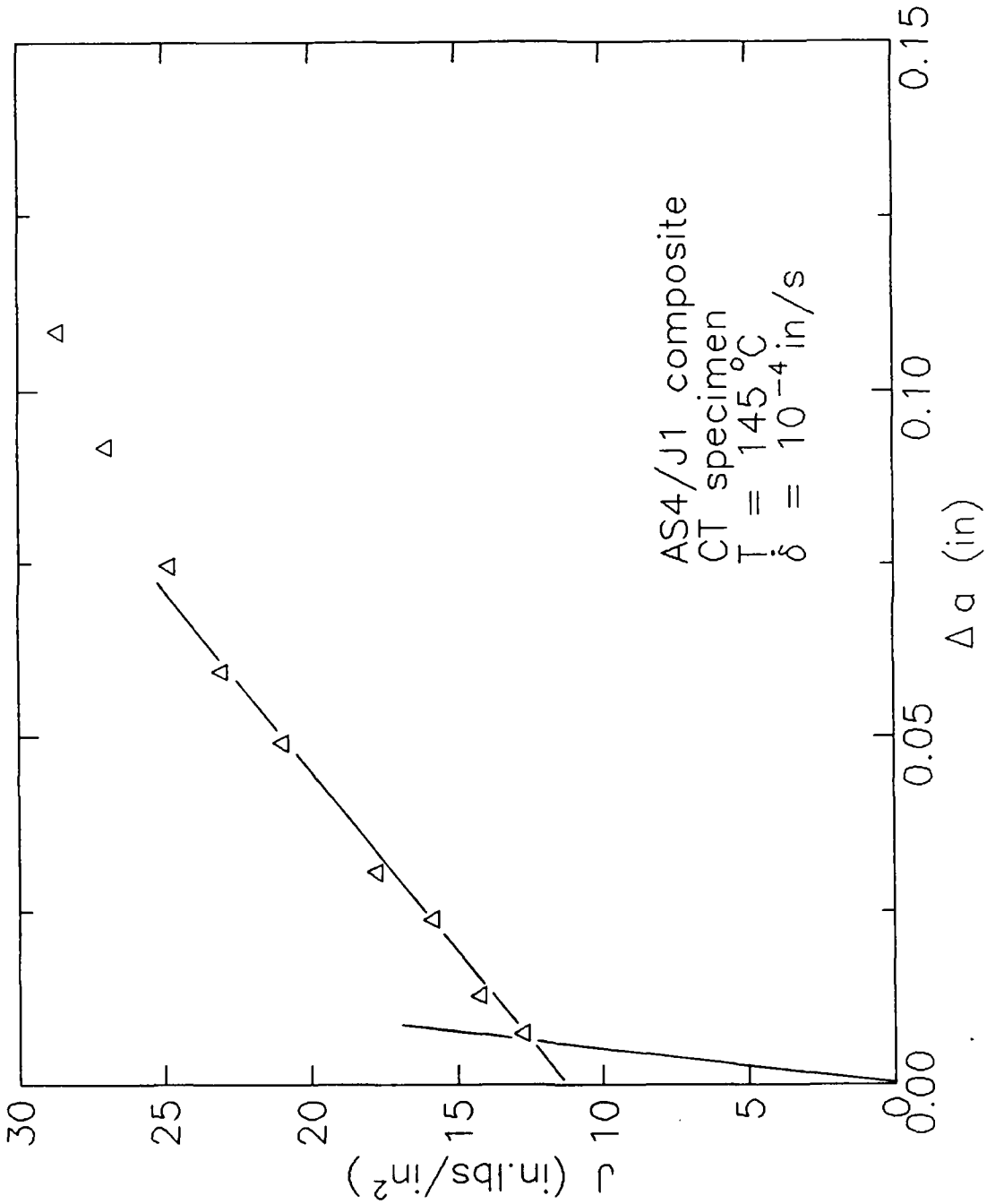

Fig. 7(c) J vs. Δa for AS4/J1 Composite. (T = 145°C)

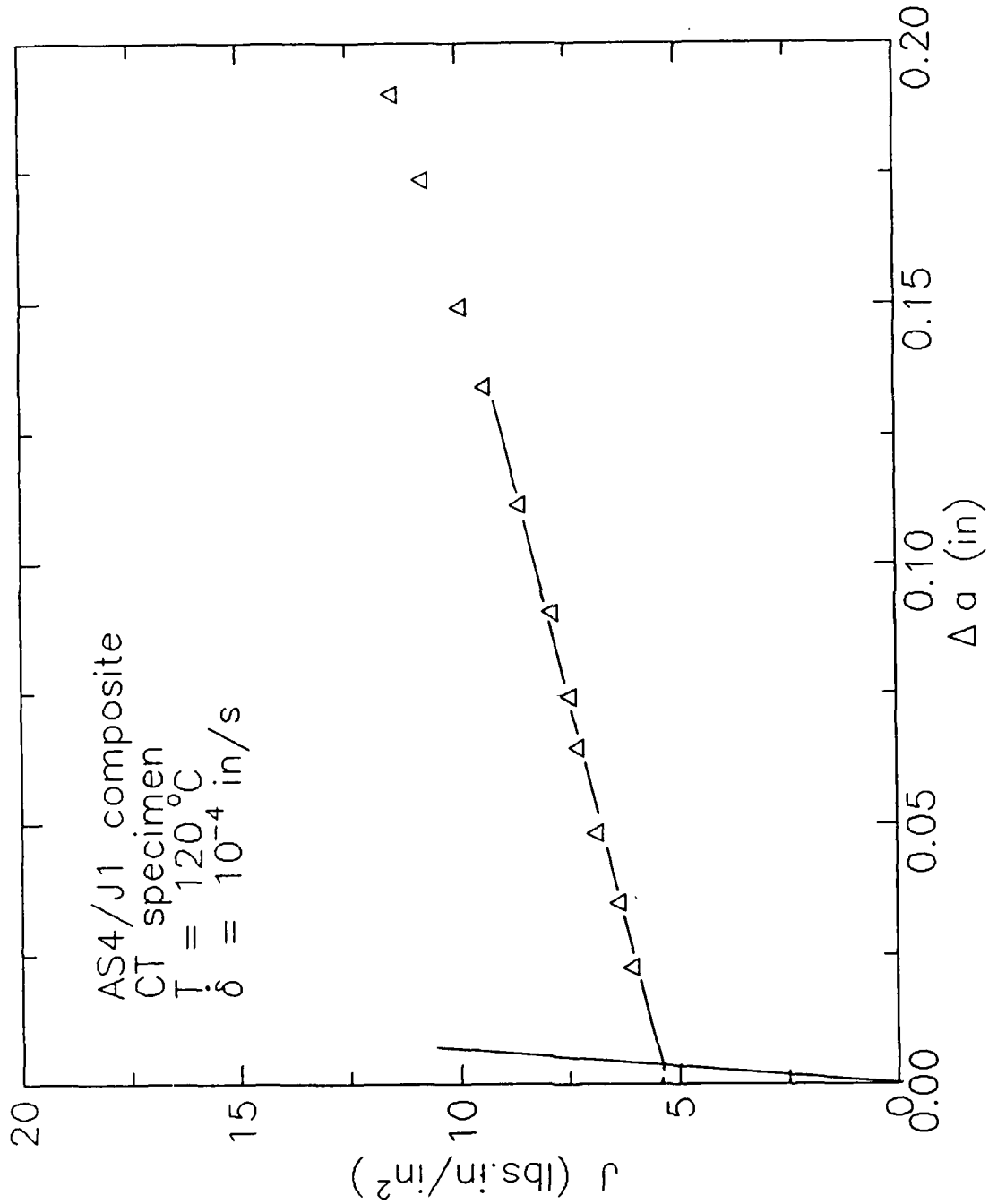

Fig. 7(d) J vs. Δa for AS4/J1 Composite. (T = 120°C)

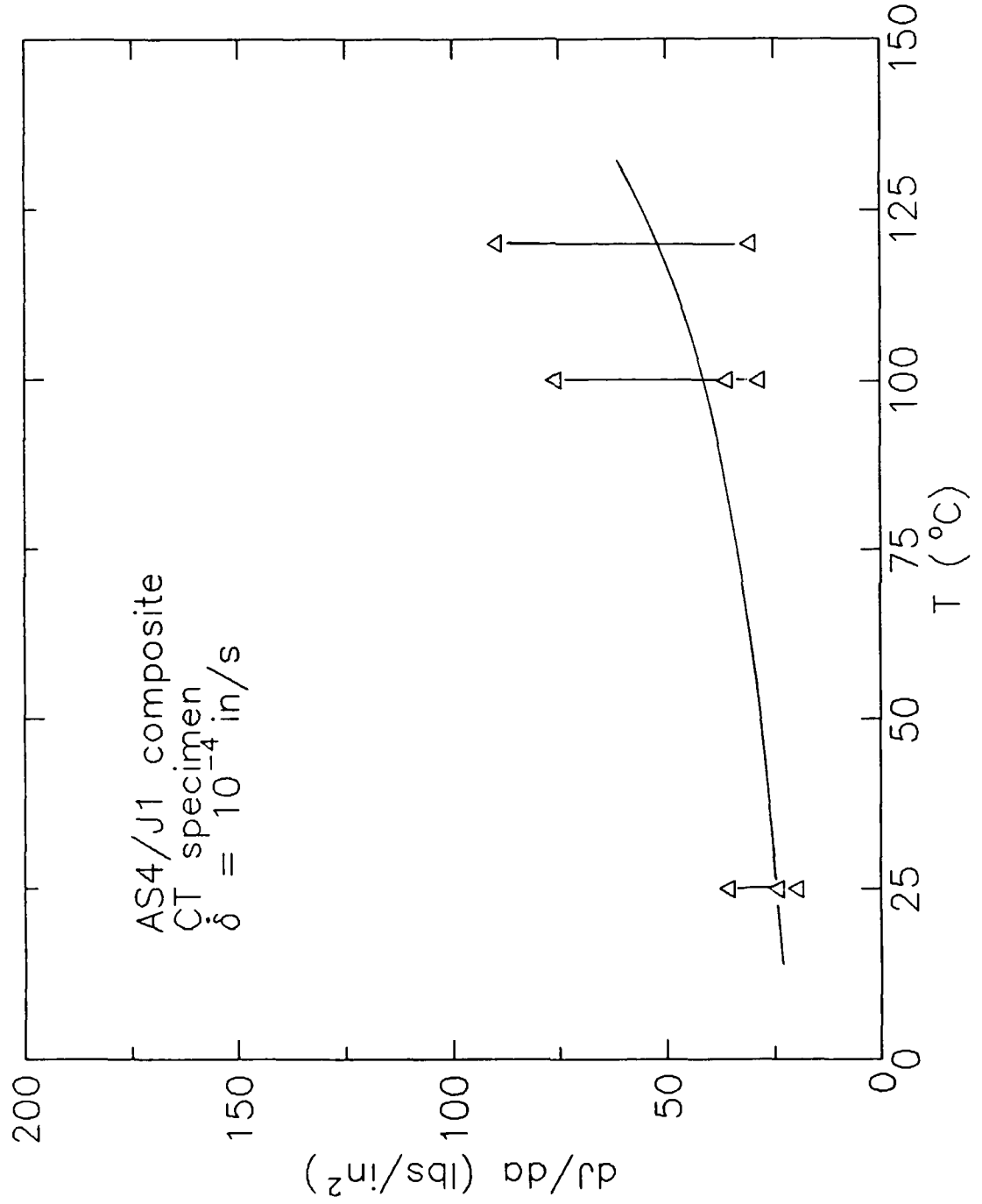

Fig. 8 Stable Crack Growth Resistance (dJ/da) vs. Temperature T for AS4/J1 Composite.

Fig. 9(a) SEM Microograph of Fracture Surface of AS4/J1 Composite at T = 25°C.

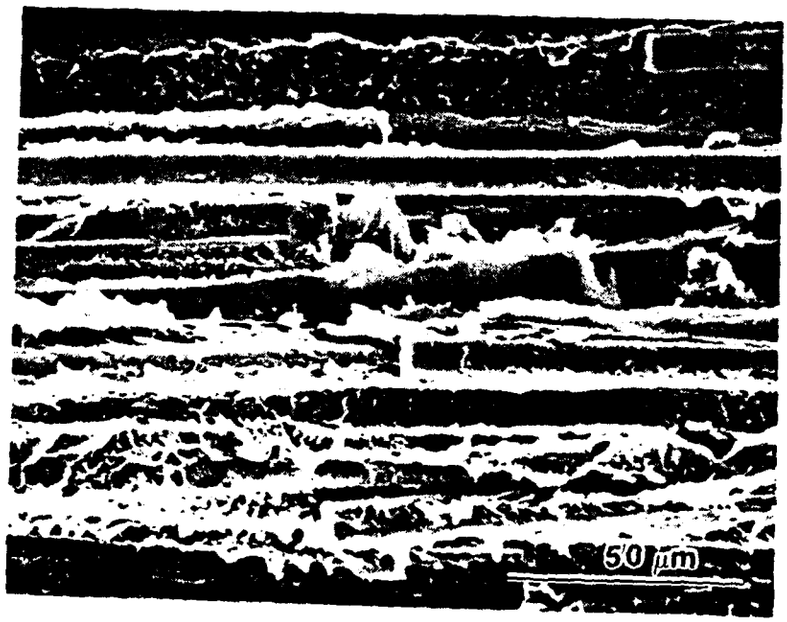

Fig. 9(b) SEM Micrograph of Fracture Surface of AS4/J1 Composite at T = 100°C.

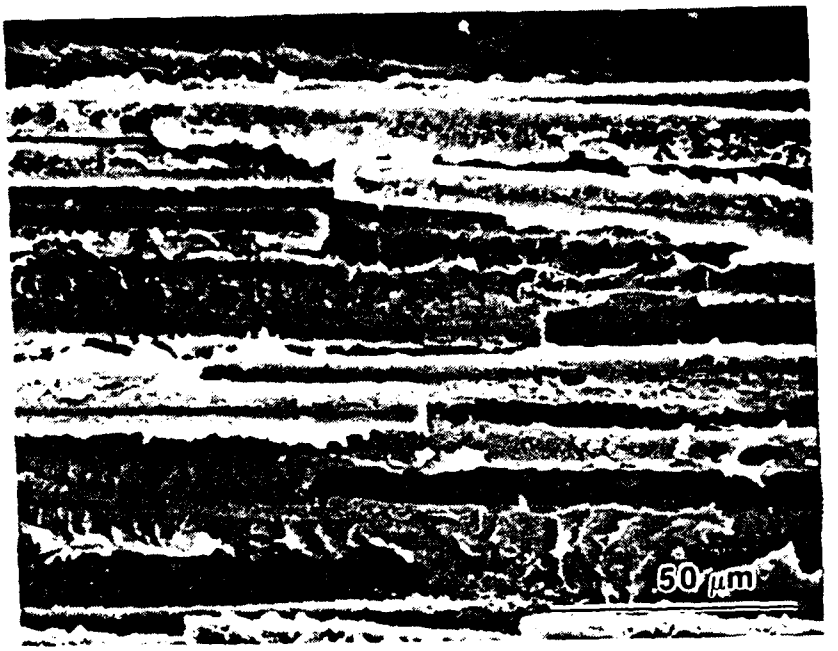

Fig. 9(c) SEM Micrograph of Fracture Surface of AS4/J1 Composite at T = 120°C.

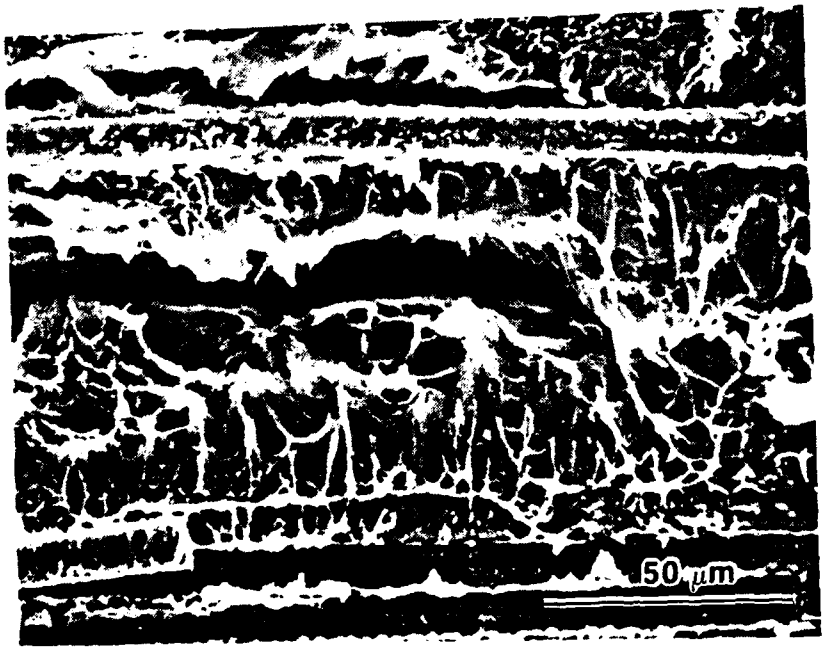

Fig. 9(d) SEM Micrograph of Fracture Surface of AS4/J1 Composite at T = 145°C.