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FINAL REPORT

FRACTURING CHARACTERISTICS OF ADHESIVE JOINTS
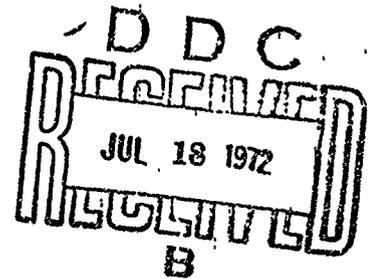
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NAVAL AIR SYSTEMS COMMAND
Washington, D. C. 20360

By

Sheldon Mostovoy and E. J. Ripling

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13. ABSTRACT

This report, written in a number of sections, is one in a continuing series on the fracturing characteristics of adhesive joints. Earlier studies in this series were concerned with stress corrosion cracking in room temperature water. These studies were extended during this period to include cracking in elevated temperature water. In addition, the effect of testing temperatures down to -321°F on the toughness of joints was also evaluated. Two types of dynamic effects were also studied: first, the effect on toughness of loading at rates up to those that cause the crack to approach its critical velocity, and second, corrosion fatigue of joints in a water environment.

Ia

14. KEY WORDS:	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Adhesives						
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Fracture Appearance						
Fracture Toughness						
Primers						
Stress Corrosion Cracking						
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**Materials
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TABLE OF CONTENTS

TABLE OF CONTENTS, Continued

TABLE OF CONTENTS

Materials
Research
Laboratory,
Inc.

TABLE OF CONTENTS, Continued

LIST OF TABLES

Materials
Research
Laboratory,
Inc.

LIST OF ILLUSTRATIONS

FOREWORD

This report covers the period March 1, 1971 to February 1, 1972. It is written in four sections two of which, the effect of test temperature and loading rate, are continuations of earlier programs. The effect of primer and temperature on stress corrosion cracking and a discussion of fatigue and corrosion fatigue are new areas undertaken during this period.

The authors gratefully acknowledge the many helpful comments of C. Bersch, Naval Air Systems Command. Mr. Paul Henderson not only prepared the graphs and line drawings for this report, but also carried out much of the experimental work for this program.

INTRODUCTION

Defining the load carrying capacity of adhesive joints, or of any structural elements, requires that they be tested over the range of conditions expected in service. If failure is assumed to occur by fracturing under unidirectional opening mode loading, these service conditions are load-time profile, the temperature and environment. The first of these, the load-time profile, includes the dynamic effect of loading, and repeated loading, as well as the static effect of time-at-load for sustained loading.

Of course, the influence of each of these cannot be considered separately. For example, a water environment would be expected to have a different influence at high and low temperature, under static sustained loading, while water may have essentially no effect at high or low temperatures under the action of a single high rate load.

Earlier programs on the evaluation of adhesive joints were concerned with the effect of long-time room temperature static loading, and especially how this was affected by water in the environment, i.e. stress-corrosion cracking. Over the past year, this work on static loading has been expanded to include the effect of elevated temperature water.

In addition to evaluating the effects of temperature on stress-corrosion cracking, the study of the effect of testing temperature on the plane strain fracture toughness was also extended to temperatures as low as -321°F and up to the post-cure temperature for four simple adhesive systems and one commercial system designated B-2 (a rubber modified, filled and "B" staged structural film adhesive typically used for bonding aircraft honeycomb).

Two types of dynamic effects were also studied; one, the effect of loading rate, over a range that varied from essentially static up to rates that approach the critical velocity, and two, a preliminary study of corrosion fatigue.

Although these studies are not as yet complete, some tentative conclusions are possible:

1. Those adhesive systems that appear to be insensitive to stress-corrosion cracking in water at room temperature do exhibit such cracking at elevated temperatures, i. e. above approximately 130°F.

2. The simple two-component adhesive systems undergo a toughness minimum in the vicinity of room temperature, or somewhat above. At temperatures as low as -320°F and as high as the post-cure temperature, the toughness is increased. The single commercial adhesive, B-2, displayed the opposite behavior. It had a maximum toughness near room temperature, and the toughness decreased at very high, and very low, temperatures.

3. The effect of very fast loading rates was again relatively simple for the two-component adhesive systems, but not for the commercial one. For the former, increased loading rate caused no significant changes in toughness until the rate was within an order or less of the critical value. Further increases in rate within this range caused a very rapid increase in toughness. Hence, a joint that is safe on slow loading would always

be safe on rapid loading. For the commercial sys-
tems, on the other hand, there appears to be a very
narrow toughness trough at cracking rates just less
than those which cause the rapid rise as the velocity
approaches its critical value. Hence, these adhe-
sives when designed for slow loading may not be safe
under rapid loading.

4. Some preliminary tests were also made to evaluate
the corrosion fatigue of joints. Three adhesives
were selected: a simple system with a primer, a
simple system (that did not exhibit stress corrosion
cracking at room temperature) without a primer, and
the commercial film adhesive. The first of these did
not exhibit slow cracking at applied crack extension
forces below the critical value. The second one cracked
at the interface on repeated loading and the third cracked
cohesively at the center of the bond. Earlier studies
had shown that the toughness of joints which cracked with
such a morphology was improved by the presence of
water. The presence of water during corrosion fatigue
was also found to improve rather than to harm the latter
joint's resistance to fatigue crack extension.

Section I

THE TEMPERATURE-TOUGHNESS PROFILE
FOR FIVE ADHESIVE SYSTEMS

During the previous contract period a study was started on the effect of testing temperature on the toughness of adhesive joints (1). This study was continued during the past year primarily to extend the range of testing temperatures down to $-300^{\circ}F$. In addition, another adhesive system was added to the program.

At each testing temperature both the initiation toughness, $\mathscr{k}_{IC}$, and arrest toughness, $\mathscr{k}_{IA}$, were measured.

MATERIALS AND PROCEDURE

The following adhesives were used in this program:

1. Ten parts per hundred of resin (PHR) of TEPA (tetraethylene pentamine) in Dow epoxy resin (DER) 332, post-cured at $180^{\circ}F$ for five hours, and designated 332/10T/180.

2. 12.5 PHR of TEPA in DER 332 resin, post-cured at $270^{\circ}F$ for five hours, and designated 332/12.5T/270.

3. 70 PHR of HHPA (hexahydrophthalic anhydride) in DER 332 resin, post-cured at $311^{\circ}F$ for five hours, and designated 332/70H/311.

4. 14.5 PHR of MPDA (meta-phenylene diamine) in Shell 826 resin, post-cured at 340°F for five hours and designated 826/14.5M/340.

5. A rubber modified commercial adhesive designated as B-2.

The first two of these were selected because they represent a system on which a great deal of toughness information is now available. The 332/10T/180 is typical of a rate insensitive adhesive within this system, and the 332/12.5T/270 of a rate sensitive adhesive.

The 332/70H/311 again is an adhesive on which a considerable amount of data is available, and of ˌrs an opportunity to compare an amine and anhydride hardener.

The 826/14.5M/340, unlike the first and second adhesive, is a high temperature curing amine, and is one that has the practical advantage of being for resistant to room temperature stress corrosion cracking, while the other two are not.

The B-2 adhesive was selected as being representative of systems presently being used for bonding of commercial aircraft sections.

The method for preparing specimens and carrying out tests was identical to that previously described (1, 2). Again 2024-T351 aluminum was used as the adherend, and these specimens tapered to have an m value of 90 in.$^{-1}$. The bonds for the poured adhesives were five mils thick, and for the commercial B-2 adhesive, 10 mils thick as a consequence of using the recommended bonding pressure.

RESULTS AND DISCUSSION

SIMPLE SYSTEMS

Fracture Toughness

Figs. I-1 through I-4 show the effect of test temperature on both ℓ_{IC} and ℓ_{IA} for the four simple systems while Fig. I-5 is the toughness-temperature curve of the commercial film adhesive, B-2. Initiation values, ℓ_{IC}, are shown as circles while the arrest values, ℓ_{IA}, are indicated by X's below the ℓ_{IC} points. For the simple systems (Figs. I-1 through I-4) the degree of rate sensitivity, as measured by the differences between initiation and arrest toughness, is largely a function of test temperature. For example, in Figs. I-1 and I-2 the two adhesive materials which were classified as "rate sensitive" and "rate insensitive", respectively, at room temperature would reverse their classification at about 140°F.

For materials described in Figs. I-1 through I-4 the test temperature substantially affected both ℓ_{IC} and the degree of rate sensitivity. Rate sensitivity was a minimum at room temperature or slightly above. The effect of temperature on arrest toughness, in general, followed the same trend as ℓ_{IC} but was much less pronounced. The single exception to this occurred for the 332/70H/311 adhesive at high temperatures, Fig. 3, where ℓ_{IC} increased rapidly while ℓ_{IA} decreased slightly with increasing temperature. For one adhesive, 826/14.5M/330, there was very little effect of temperature on either ℓ_{IC} or ℓ_{IA}. Initiation toughness at very low temperatures, i.e. at -300°F, was surprisingly high for all the simple systems, and these frequently exhibit their lowest toughness near room temperature

Fracture Appearance

Fracture appearance of these simple adhesive systems at low temperature showed a rough "washboard" appearance and, as the temperature was increased, fracture morphology became smooth, center-of-bond, and at still higher temperatures where the toughness recovered, fracture became mottled, as previously described ([3]). The high toughness at high and low temperatures is associated with this roughness.

COMMERCIAL ADHESIVE

Fracture Toughness

Toughness of the B-2 commercial film adhesive, Fig. I-5, was considerably different. This material showed a toughness maximum at about room temperature (R. T.) that was more than order of magnitude higher than the simple systems. In addition, the material was rate insensitive and crack extension occurred at a constant k_{IC}. The load-displacement (P-Δ) curve of this material did not show the abrupt transition from a stationary crack to a moving crack that is typical of the simple adhesive systems. Instead, the P-Δ record indicated that crack growth began at a k_i value about 20 percent less than the steady state value of k_{IC}. (Hence these values cannot be properly identified as k_{IC}, and in the vicinity of the peak might be of the order of 20 percent less than the reported values.) At test temperatures above 75°F the k_{IC} value decreased to a value of about 4.0 lbs/in. at 350°F, the post-cure temperature. In the temperature range between -25 and -100°F there was a sharp loss in toughness to a value of 1.0 lbs/in. at test temperatures below -100°F. This "transition range" where the very tough material became comparatively brittle was also accompanied by a change in the

P-Δ record from a rate insensitive to rate sensitive behavior. In the low temperature brittle range the material showed distinct initiation and arrest values and these are plotted using X's for the arrests as was done for the simpler systems.

A comparison of the B-2 system with initiation toughness of the simple adhesives is shown in Fig. I-6 and with arrest toughness in Fig. I-7. Clearly, at test temperatures between -50°F and 250°F the commercial system is far superior to the simpler materials but at the low temperatures the commercial system is no better than the others. At the increased test temperatures the elevated-temperature-curing simple systems rise quite rapidly and begin to approach the declining B-2 $\mathcal{K}_{IC}$ value.

Fracture Appearance

Fracture appearance of the B-2 material at elevated temperatures, Fig. I-8, shows a transition from the rough center of bond (CoB) behavior at room temperature to a partial interface fracture at elevated temperature. Fracture appearance of the simple systems remained smooth CoB as test temperature was increased but these also showed some partial interface (IF) or "spongy" fracturing as the test temperature approached the post-cure temperature. The most obvious change in the smooth CoB fracturing with increasing temperature was the evidence of an increased amount of plastic flow (e.g. transverse rippling).

Fracture appearance for the B-2 adhesive below room temperature (R. T.) was quite similar to the rough CoB room temperature fracturing until test temperatures below -50°F were reached. At this temperature and below, fracture tended to be smooth CoB and similar in

appearance to the simple systems at R. T. and below. At extremely low temperatures, -290°F, the sharp up-turn in toughness for the simple systems was marked by a distinct roughening of the fracture appearance denoted "washboard".

CONCLUSIONS

The effect of temperature on toughness of both simple and commercial adhesive systems might be summarized as follows:

1. For all the simple adhesive systems tested there appears to be a broad minimum in initiation toughness between -300°F and the post-cure temperature. In all cases, $\mathcal{K}_{IC}$ increased rapidly in the vicinity of the post-cure temperature. Toughness at very low temperatures were also surprisingly high.

   The arrest toughness, $\mathcal{K}_{IA}$, was less dependent on temperature than $\mathcal{K}_{IC}$. In some cases, the former showed a broad minimum in the toughness-temperature profile similar to $\mathcal{K}_{IC}$, in others it decreased continuously, or remained unchanged as temperature increased.

2. Unlike the simple adhesives, the toughness of the B-2 adhesive is maximized at room temperature, and its absolute value of $\mathcal{K}_{IC}$ is at least an order higher than that of the two component systems. At temperatures below about -50°F the plasticizer in the B-2 ceases to operate. This results in a "transition

temperature" behavior with a severe toughness loss
such that both toughness and fracture appearance for
this adhesive are similar to the simple systems.

3. A rough, partially IF ("spongy") fracture appearance
    is associated with high toughness in both the commer-
    cial plasticized epoxy system and the simple two-com-
    ponent adhesive.   At test temperatures below $-150^{o}F$,
    where the toughness of all systems increases, this in-
    crease is accompanied by a roughening of the fracture
    surface ("washboard").   Test temperatures near post-
    cure were marked by some IF fracturing irrespective
    of the measured toughness.

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3. S. Mostovoy and E. J. Ripling, "Effect of Temperature on the Fracture Toughness and Stress Corrosion Cracking of Adhesives", to be published.
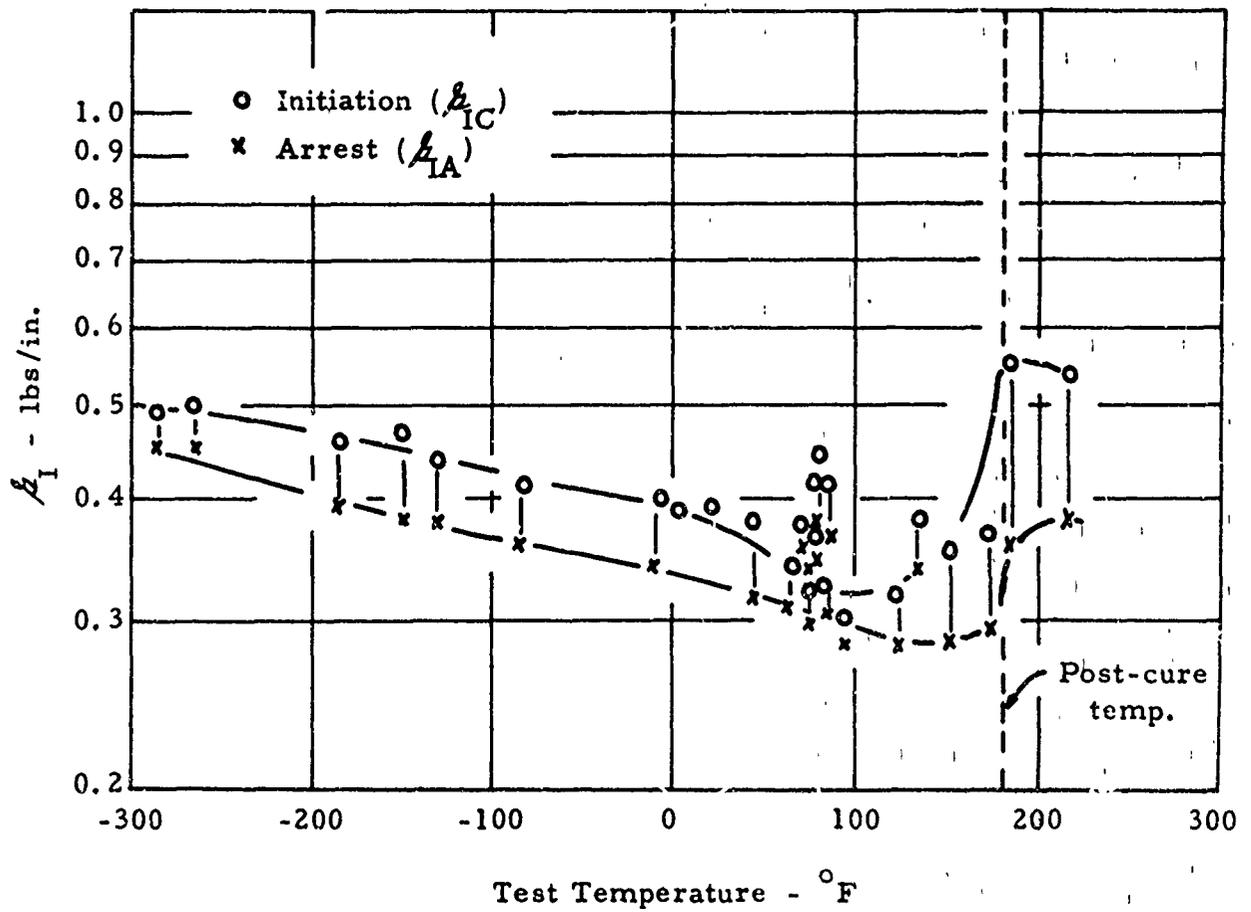
Fig. I-1   Effect of Temperature on Toughness of 5 mil Bonded
332/10T/180 Adhesive Specimens (Crosshead rate:
1.0"/min.).
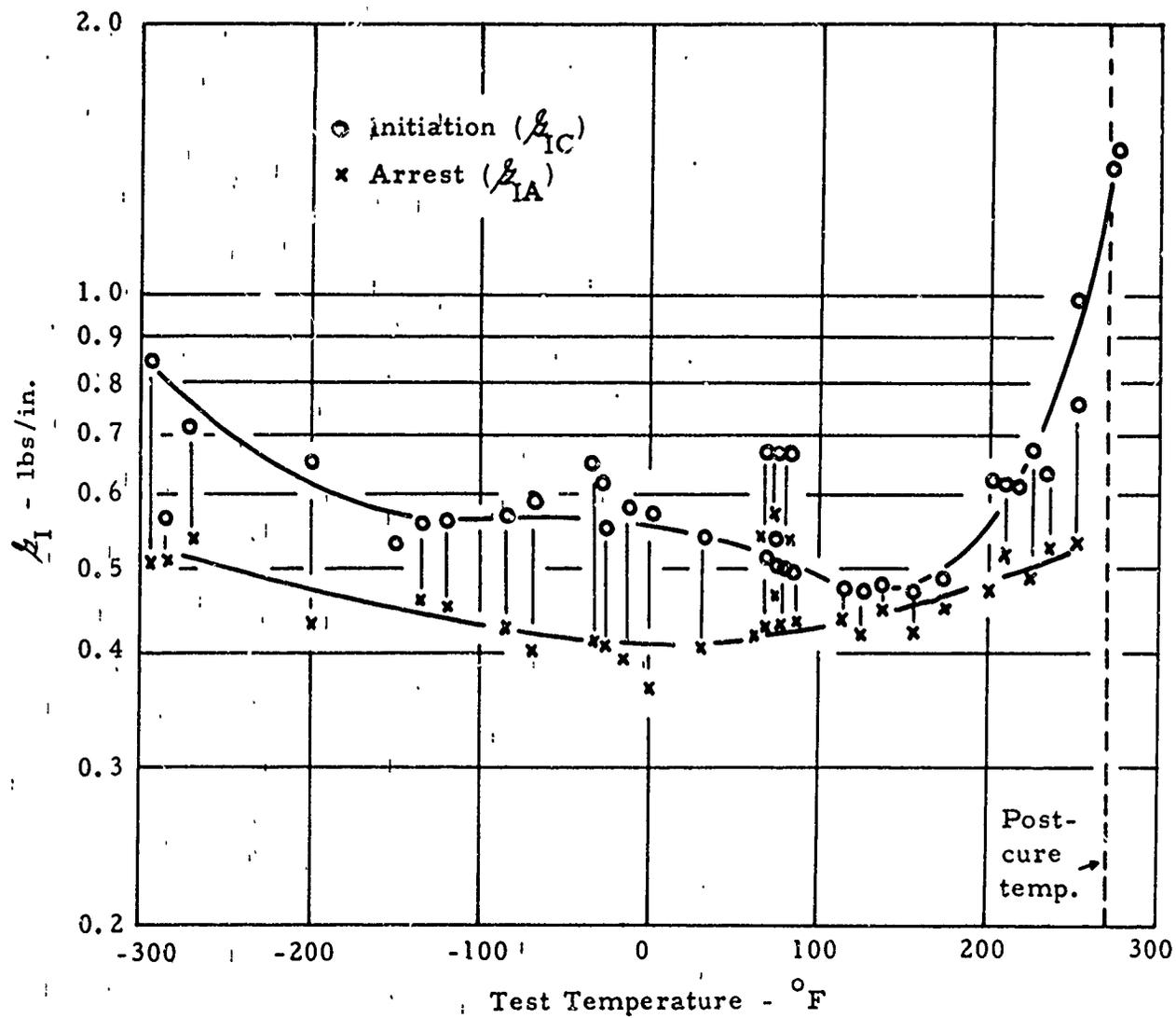
Fig. I-2   Effect of Temperature on Toughness of 5 mil Bonded 332/
12.5T/270 Adhesive Specimens (Crosshead rate: 1.0"/min.).
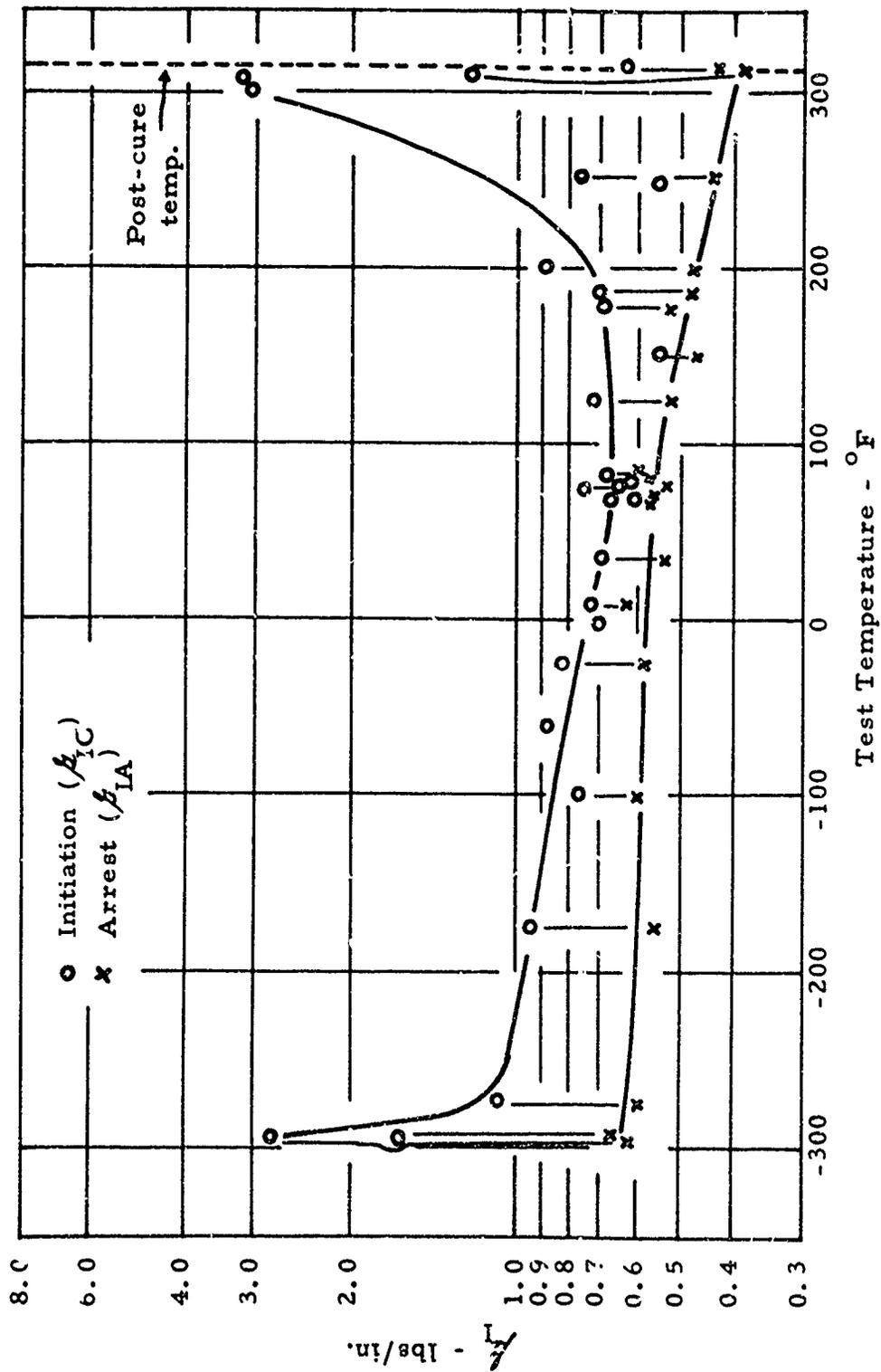
Fig. I-3 Effect of Temperature on Toughness of 5 mil Bonded 332/70H/311 Adhesive System (Crosshead rate 1.0"/min.).
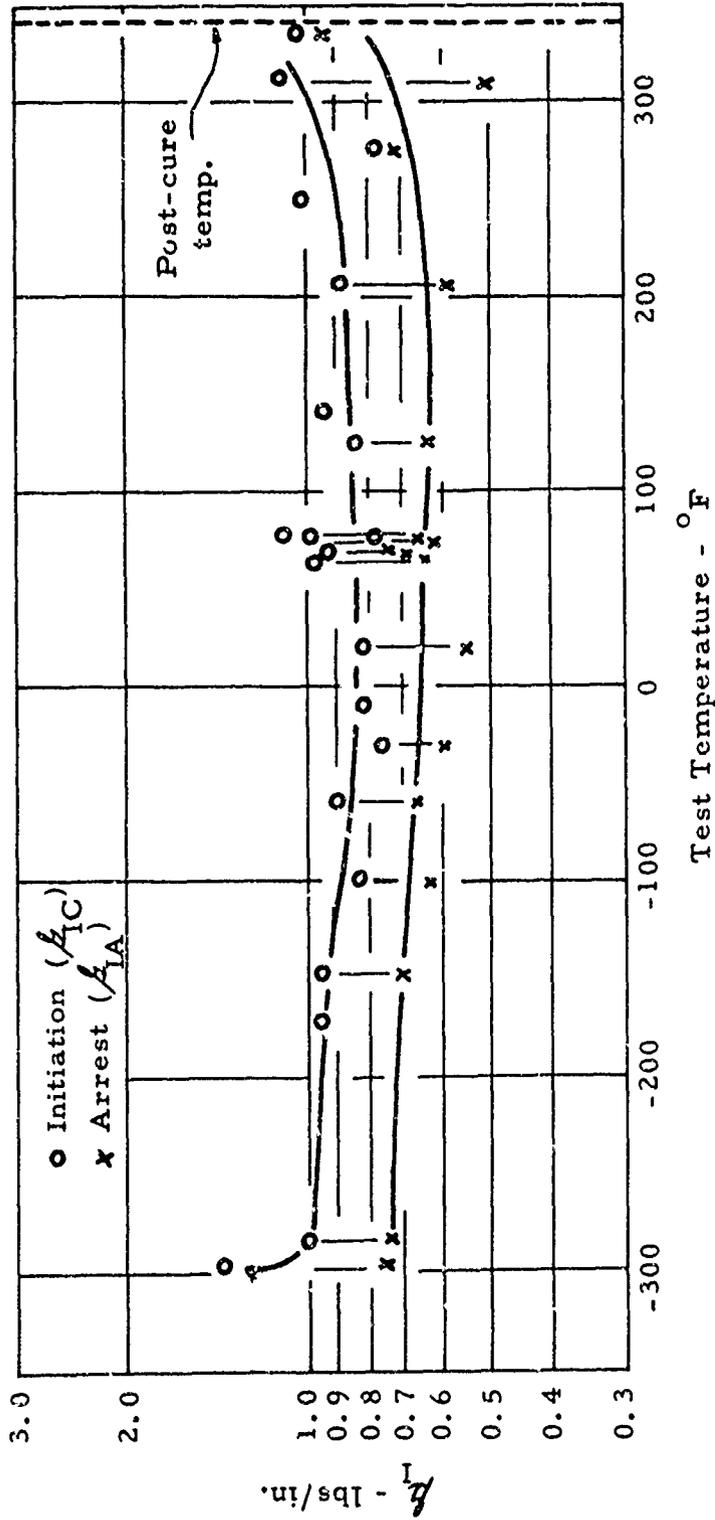
Fig. I-4   Effect of Temperature on Toughness of 5 mil Bonded 826/14.5M/340 Adhesive Specimens (Crosshead rate: 1.0"/min.).
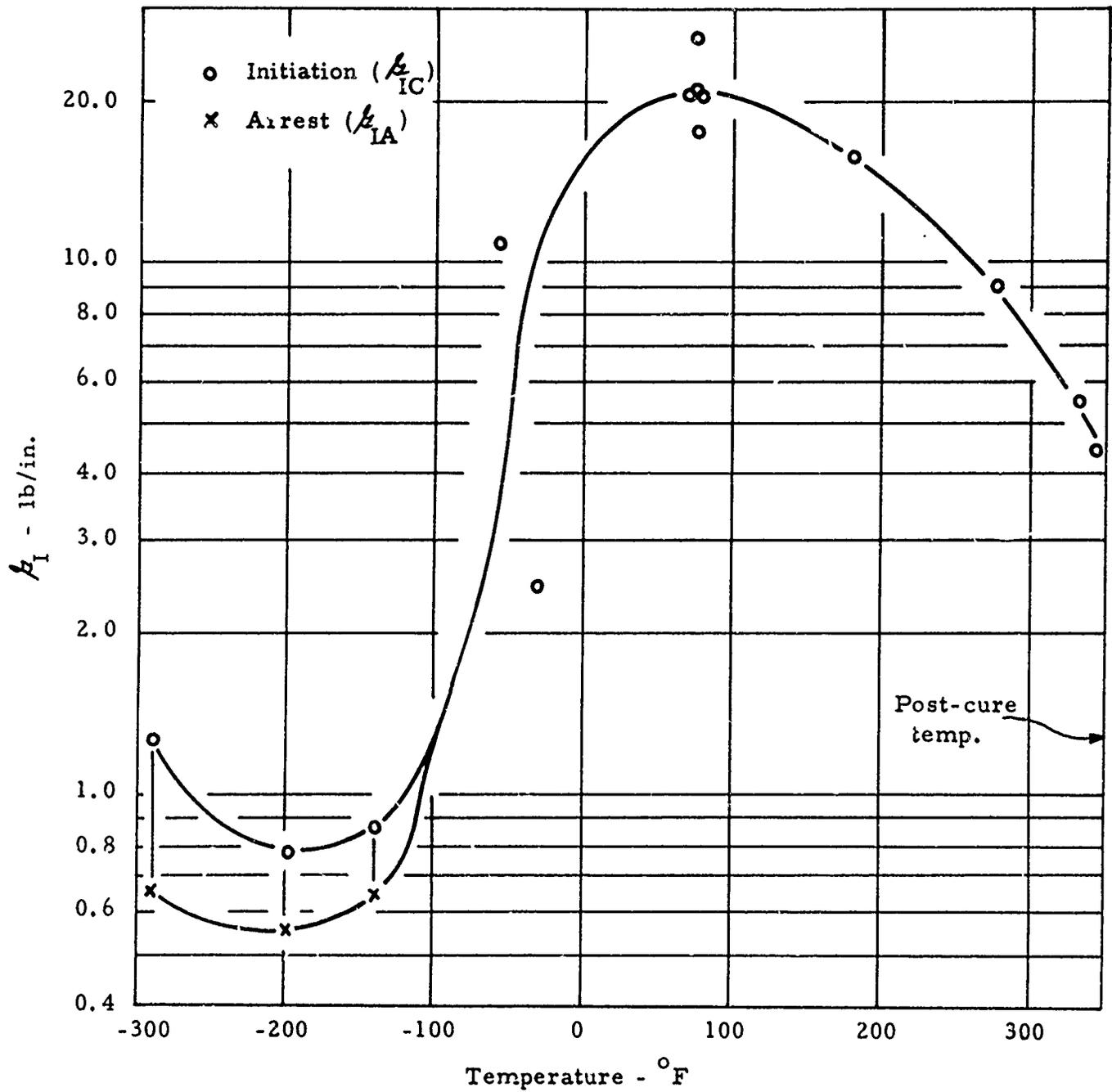
Fig. I-5   Effect of Temperature on Toughness of B-2 Adhesive.
(Crosshead rate: 1.0"/min.).
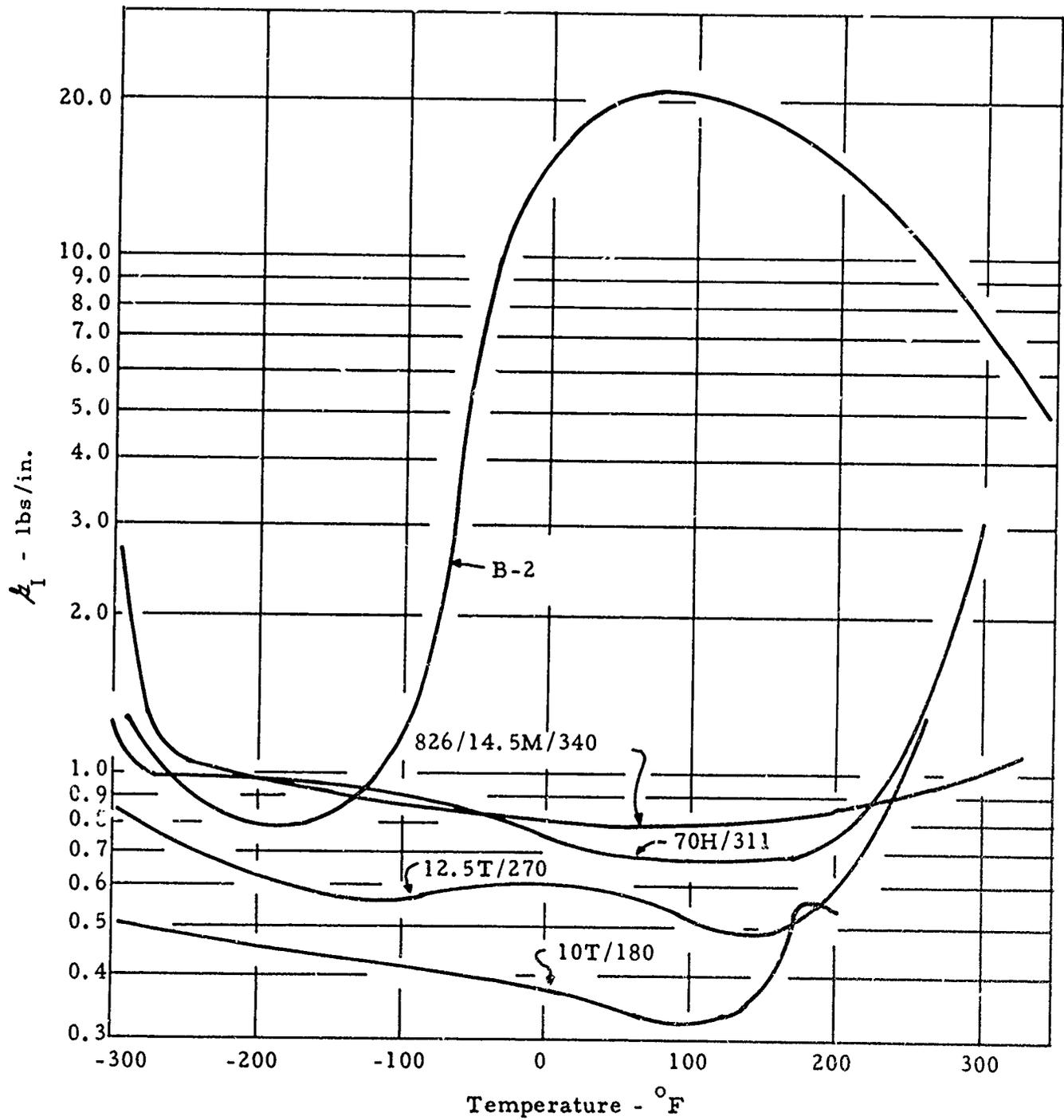
Fig. I-6   Initiation Toughness Profile for Several Adhesive Systems.
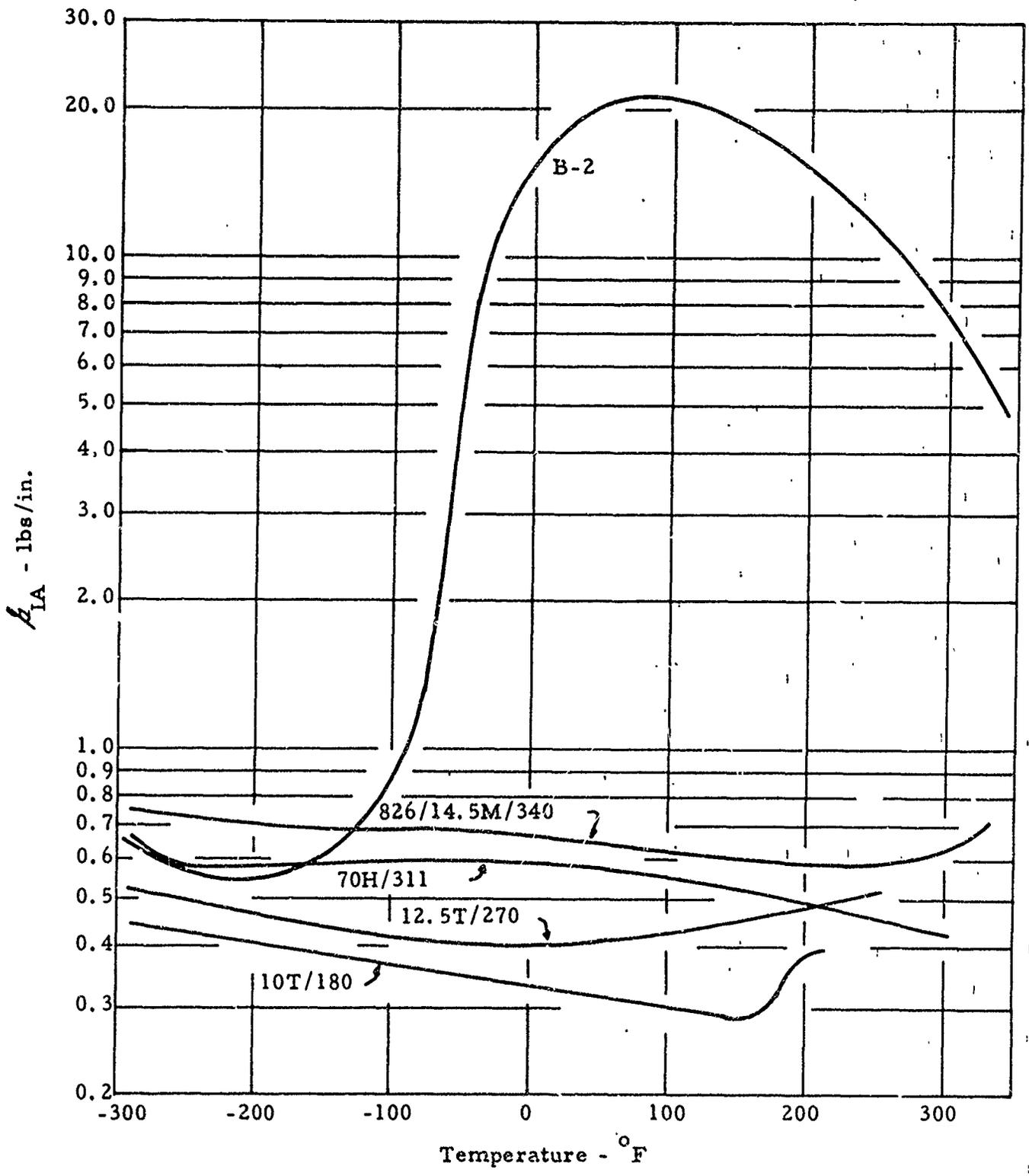
Fig. I-7   Arrest Toughness Profile for Several Adhesive Systems.

R. T. $\longleftarrow$ 275°F $\longrightarrow$ $\longleftarrow$ 335°F

Crack Growth Direction $\longrightarrow$

R. T. $\longrightarrow$ $\longleftarrow$ 345°F

-60°F
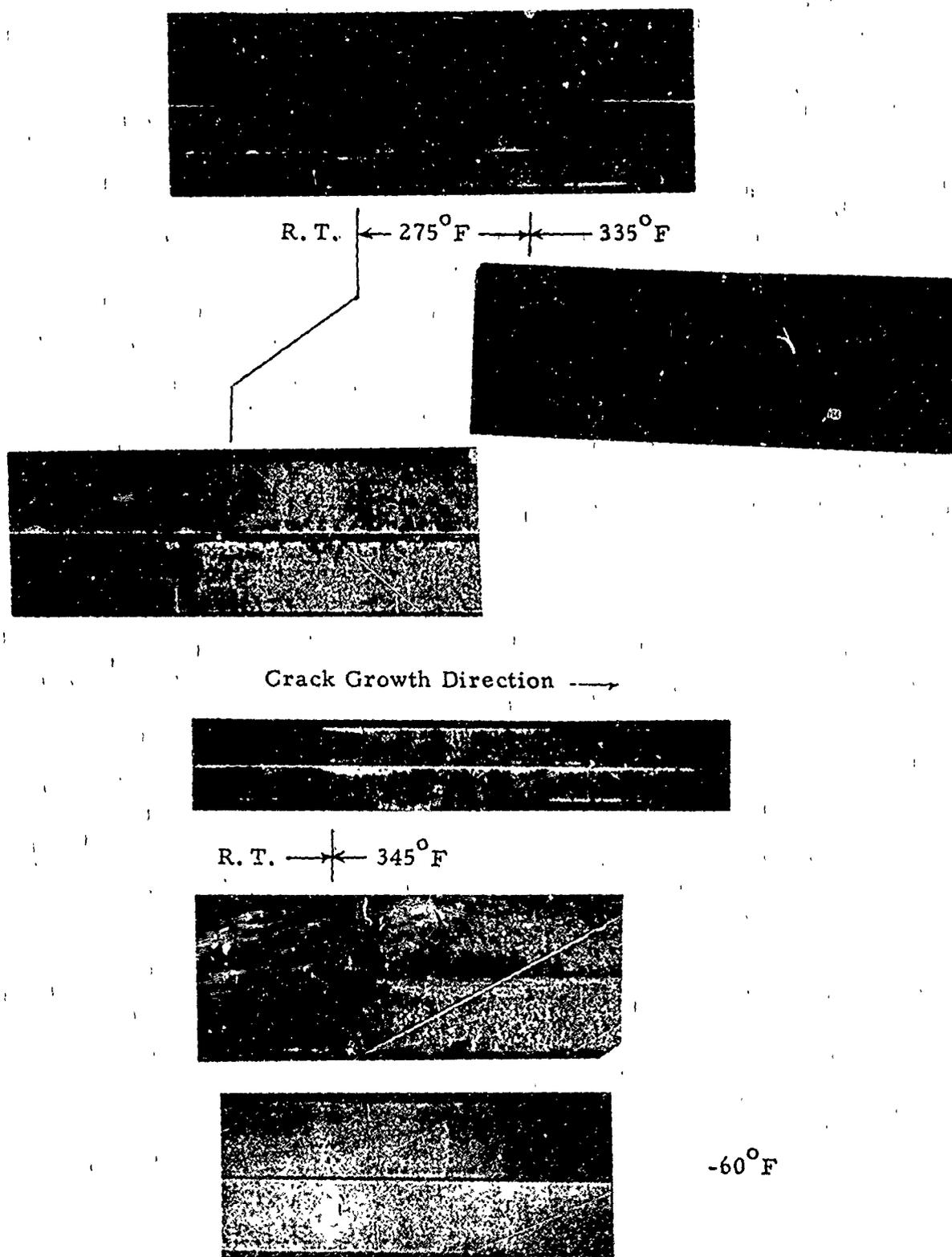
Fig. I-8   Typical Fracture Appearance of Commercial Adhesive
(B-2).

Section II

THE TOUGHNESS-LOADING RATE PROFILE FOR
TWO SIMPLE AND ONE COMMERCIAL ADHESIVE SYSTEM

INTRODUCTION

The first section of this report described the effect of tempera-
ture on toughness. This section is concerned with the influence of
loading, or displacement, rate and crack velocity on toughness. To
relate cross-head velocity or displacement rates, $\dot{\Delta}$, to crack rates,
$\dot{a}$, it is necessary to examine the shape of the load-displacement (P-Δ)
record. As pointed out in earlier reports, two types of cracking be-
haviors are found with zero-K-gradient specimens: the crack may ex-
tend at a rate dictated by the cross-head motion so that the crack initiates
and propagates at a constant load. P-Δ curves having this character-
istic are labelled "flat", and materials that show this behavior are
"rate insensitive". For these, an increase in cross-head rate (and
thus cracking rate) resulted in an increased toughness (or increased
load). In the second type the crack outran the cross-head and the load
dropped rapidly to the value of load necessary to arrest the fast moving
crack from the load required for initiation. Such behavior is denoted
"peaked", from the shape of the P-Δ record, and the behavior is denoted
"rate sensitive". Measurement of cracking rates for "peaked" behavior
indicated that toughness increased as rate increased. Rates of the order
of 100 ft/sec. at toughness values of 1.5 lbs/in. were noted on some ad-
hesive formulations. This compared with toughness values of 0.3 lbs/
in. for cracks driven at about 0.1 ft/sec for $\frac{1}{2}$ inch thick aluminum
adherend specimens having a shape factor of m = 90 in.$^{-1}$. This corres-
ponds to a cross-head rate, $\dot{\Delta}$, of 1.0 in./min.

The present study was undertaken for the following specific purposes:

1. To compare the toughness of the simple adhesive systems with that of commercial adhesive as a function of increasing rate.

2. To extend the cracking rates on all systems tested to the vicinity of the limiting velocity (e.g. $C_o/4$ or approximately 1250 ft/sec in bulk epoxy of 0.5×10^6 modulus).

3. To define differences in "rate sensitive" ("peaked") and "rate insensitive" ("flat") adhesive material at high rates.

4. To correlate fracture morphology with cracking rate to obtain the toughness associated with fracture appearance in adhesives where the toughness during crack growth was not known.

MATERIAL AND PROCEDURES

Material

Three of the five adhesives used in the temperature study were used in this program. These were the two simple systems 332/10T/ 180, 332/12.5T/270 and the commercial system B-2. The specimen preparation procedure was also identical with that given above.

Procedure

In earlier work cracking rate data was obtained using an external load cell and displacement transducer. Since data were to be collected

at very high rates on this program, the use of external load cells was
not feasible so that strain gages were mounted directly on one of the
aluminum adherends.   This procedure minimized "ringing" in the load
records.   The ga · ₃ were calibrated with the external load cell at lower
than critical loads prior to conducting the test.

In order to obtain the highest cracking rates possible with a
standard low capacity (3000 lbs.) high-rate testing machi .e (maximum
cross-head rate, $\dot{\Delta}$, of 6 ft/sec.) it was also necessary to use more
steeply tapered aluminum adherends, i.e. $m = 4$ in.$^{-1}$.   For this speci-
men the expression for cracking rate is:

$$\dot{a} = (\frac{Eb}{8 P_c m'}) \dot{\Delta} = 115 \times 10^3 \frac{\dot{\Delta}}{P_c}$$

where

$\dot{a}$ = cracking rate, da/dt

E = elastic modulus of the adherends = 10^7 psi

b = adherend width

m' = 5.45 in.$^{-1}$

$\dot{\Delta}$ = cross-head displacement rate, $d\Delta/dt$

P_c = critical load at which crack extends

If the crack extends under a condition of constant load the exact value of
$\dot{a}$ can be calculated from the P-t record and a knowledge of the crack
length.   For this specimen the compliance at any crack length is

$$C = 5 \times 10^{-6} + 8.73 \times 10^{-6} (a - 1) \text{ lbs/in.}$$

But

$$C = \frac{\Delta}{P} \text{ and } C = (\frac{\dot{\Delta}}{\dot{P}})$$

$\therefore$

$$\dot{\Delta} = \dot{P}C = \dot{P} \left[5 + 8.73 (a - 1) \right] \times 10^{-6}$$

thus, to obtain the cross-head rate, $\dot{\Delta}$, the slope of the P-t curve is

measured and, using the crack length obtained after the test is completed (or from prior compliance measurements), the above equation is used to calculate it.

The value of $\underline{\dot{a}}$ based on constant load extension is the lowest value that the cracking rate can have. If the crack outruns the cross-head the calculated value of $\underline{\dot{a}}$ represents a lower bound which can be used along with the critical load for crack extension to obtain a point on the $\mathscr{G}_{IC}$ vs. $\underline{\dot{a}}$ curve.

Measurements of P-t curves were made with an oscilloscope camera set to record a single trace. Triggering was accomplished by a displacement contact which activated the scope for a single display. This mechanical triggering was necessary since at the fastest sweep speed, 2 msec/cm., the curve could not be recorded by manual or internal triggers. The test sequence was as follows:

1. Oscilloscope set for proper sensitivity and sweep frequency.

2. Specimen inserted and high rate cylinder slowly moved such that a small load was placed on the specimen.

3. Strain gage on specimen was compared with load cell in loading train to set calibration.

4. Specimen unloaded and fixed slack taken in the coupling to allow for cylinder inertia during test.

5. Camera set and opened.

6. Loading pulse triggered on cylinder.

Records obtained in this way were quite acceptable and the resultant

data was analyzed for $\dot{a}$, Δ and $\mathcal{L}_{IC}$ by the methods already described.

RESULTS AND DISCUSSION

Values of $\dot{a}$ vs. $\mathcal{L}_{IC}$, obtained as described above on 332/10T/

180 (an adhesive system always driven at rates predicted by the cross-

head) are shown in Fig. II-1. Although much of the data below 100 ft/

sec. were collected earlier, several additional points were placed in the

$\dot{a}$ range between 10 and 100 ft/sec. using $m = 4$ in.$^{-1}$ specimens. Cor-

responding $\mathcal{L}_{IC}$ values had peaks and valleys not unlike the "rate-spec-

trum" phenomena seen for steels as reported by J. Krafft at NRL. This

is in contrast to the constant or slowly rising toughness with increasing

rate seen in the $\dot{a}$ range below 10 ft/sec. Above 100 ft/sec. there is a

very rapid rise in toughness as the cracking rate approaches its limiting

value of $C_o/4$ ($\simeq$ 1250 ft/sec.). In this $\dot{a}$ range $\mathcal{L}_{IC}$ changes from

about 0.5 to 5.0 lbs/in. as $\dot{a}$ changes from 150 to 600 ft/sec.

Rate data on the 332/12.5T/270 adhesive also showed this

rapid rise in toughness in the $\dot{a}$ range above 100 ft/sec., Fib. II-2. In

fact, in this range, values of $\mathcal{L}_{IC}$ become comparable between this sys-

tem and 332/10T/180, Fig. II-3. Below 100 ft/sec. the crack outruns

the cross-head ("peaked" P-Δ records). Thus, data in this range (see

Fig. II-2) is plotted to show cross-head rates used to define the lower $\dot{a}$

limit where the crack is driven. There can be no "rate-spectrum" below

100 ft/sec. since crack always chooses to run at this velocity unless

pushed to higher rates by the test machine.

In addition to the simple system described above, a rate

curve was obtained on the B-2 commercial rubber modified epoxy film

adhesive; limited data taken on this system is shown in Fig. II-4.
Only the very highest and very lowest rates have been obtained. However, $\mathcal{A}_{IC}$ does appear to rise as $\dot{a}$ increased to 600 ft/sec. after which there is a drop off to lower levels than seen at the lowest rates. It is expected that this drop off corresponds to a change in fracture morphology; the rubber particles which blunt the crack tip at lower velocities are not as effective at high rates. Examination of the fracture surfaces does show a change in morphology from the very rough type seen at low rates to a moderately rough variety at the higher rates. The change in fracture morphology in the controlled rate tests is slight. However, the fracture surface at the back end of the most rapidly loaded specimens was smooth. At this location the crack is travelling at the highest velocity obtained to date since the specimen compliance rather than being constant is increasing rapidly. Although no toughness data accompanies it, it is likely that $\mathcal{A}_{IC}$ for this relatively smooth fracture is much lower than that measured for the other fractures shown. On the basis of this fracture appearance, and knowing that the toughness must become very high as $\dot{a}$ approaches its critical velocity, the curve shape for B-2 can be approximated as having a very narrow, deep trough at $\dot{a}$ just less than $C_o/4$.

A comparison of the rate curves for all three adhesives, Fig. II-5, illustrates that at high rates the toughness of all materials approach one another. As stated above, it is expected that if $\mathcal{A}_{IC}$ data were available for the smooth fracturing of the B-2 adhesive this approach might be closer than what is shown in the figure.

CONCLUSIONS

An expected result of this work is the rapid upturn in toughness of the simple adhesive systems with increased rate. This upturn occurred at about 100 ft/sec. Above this rate, differences in the shape of P-Δ curves and differences in toughness no longer exist, and the fracture surface of all simple systems become rough CoB (frosty) rather than the shiny CoB seen at lower rates.

The behavior of the commercial film adhesive B-2 differs from that of the simple systems. Although the fast increase in toughness is found above $\underline{\dot{a}}$ of 100 ft/sec., this is followed by a deep trough prior to the toughness obtaining its very high value at velocities associated with its critical value.
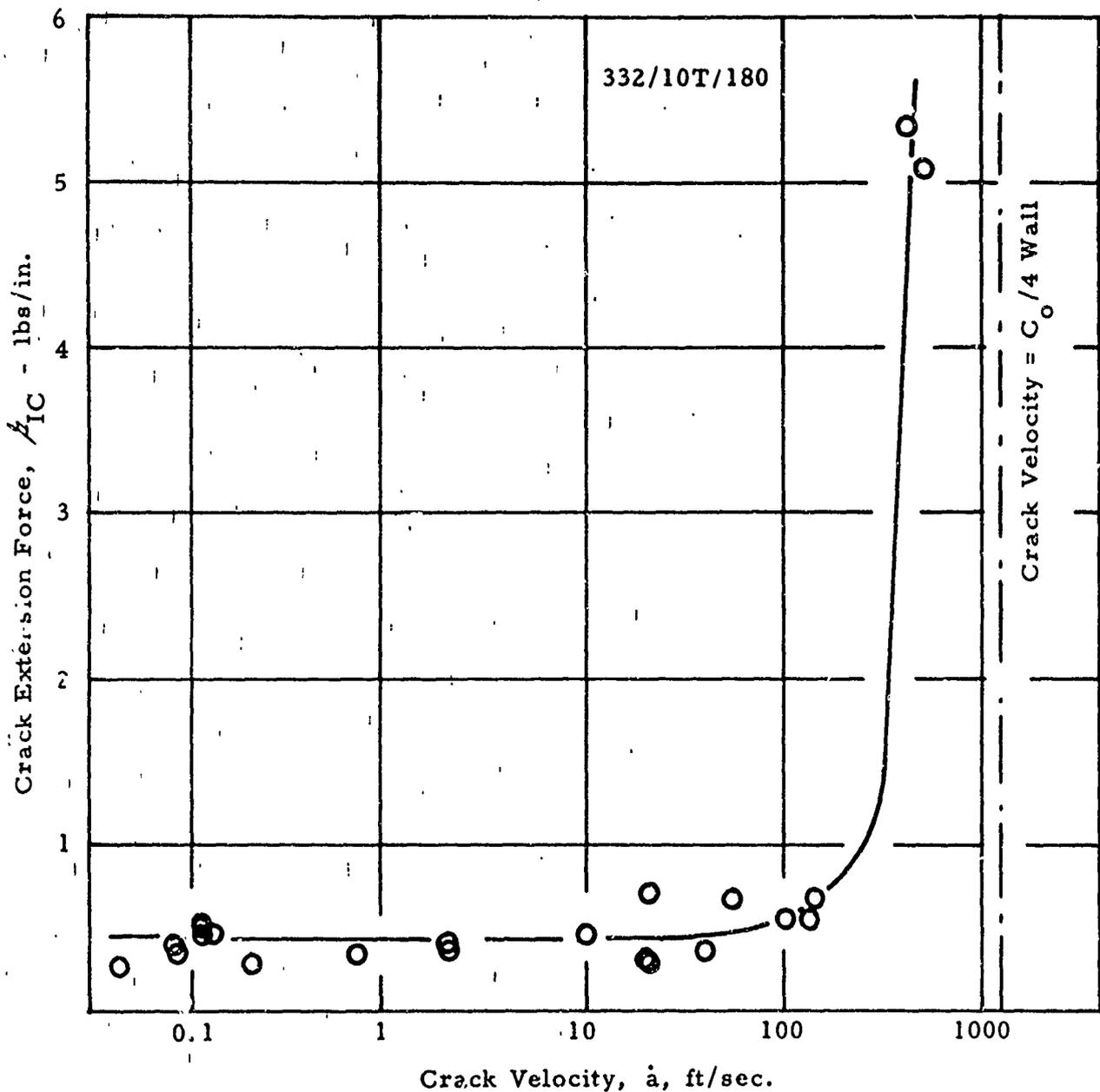
Fig. II-1   Effect of Crack Speed, ȧ, on Toughness, $\not{b}_{IC}$, for 5 mil
Bonded 332/10T/180 Adhesive.   Aluminum Adherends.
Highest Speeds Obtained with m = 4 Taper.
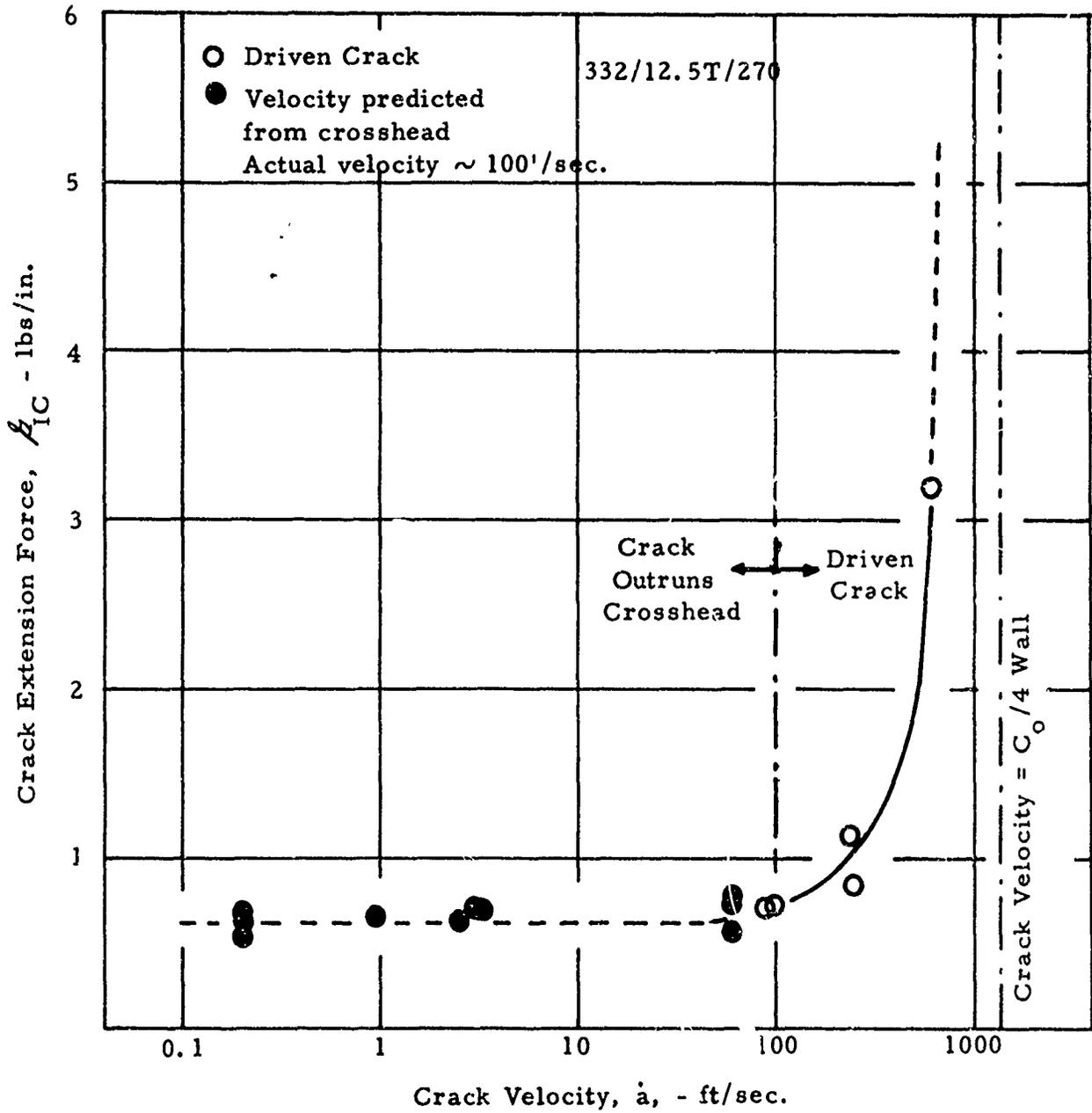
Fig. II-2 Effect of Crack Speed, à, on Toughness, $\mathscr{F}_{IC}$, for 5 mil
Bonded 332/12.5T/270 Adhesive. Aluminum Adherends.
Highest Speeds Obtained with m = 4 Taper.
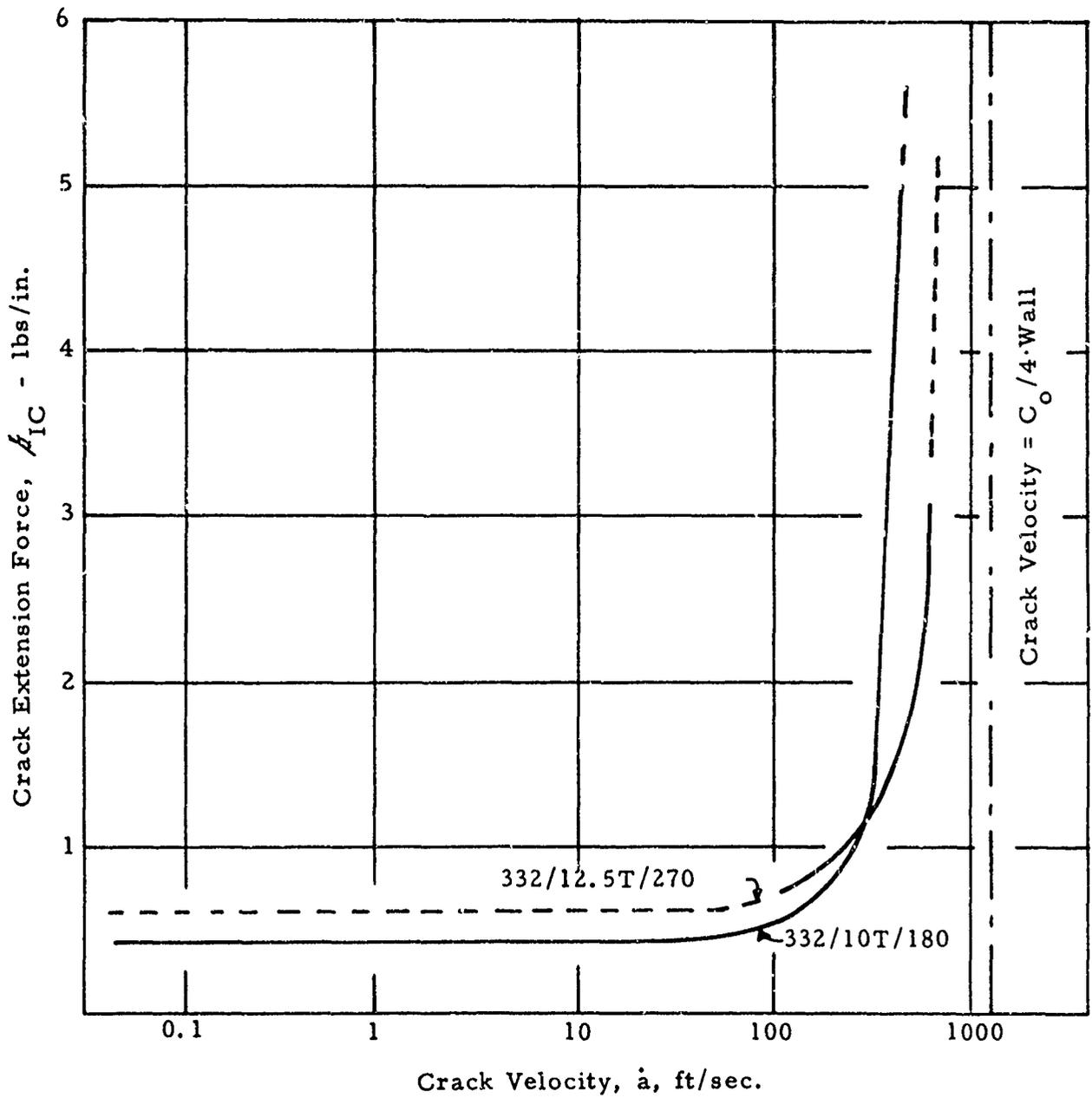
Fig. II-3   Toughness Comparison as a Function of Crack Speed for
             332/10T/180 and 332/12.5T/270 Adhesives.   Note that at
             velocities above 100 ft/sec. toughness of both systems are
             comparable.

-28-

Fig. II-4   Effect of Crack Speed, $\dot{a}$, on Toughness, $\mathcal{F}_{IC}$, for B-2 Adhesive.
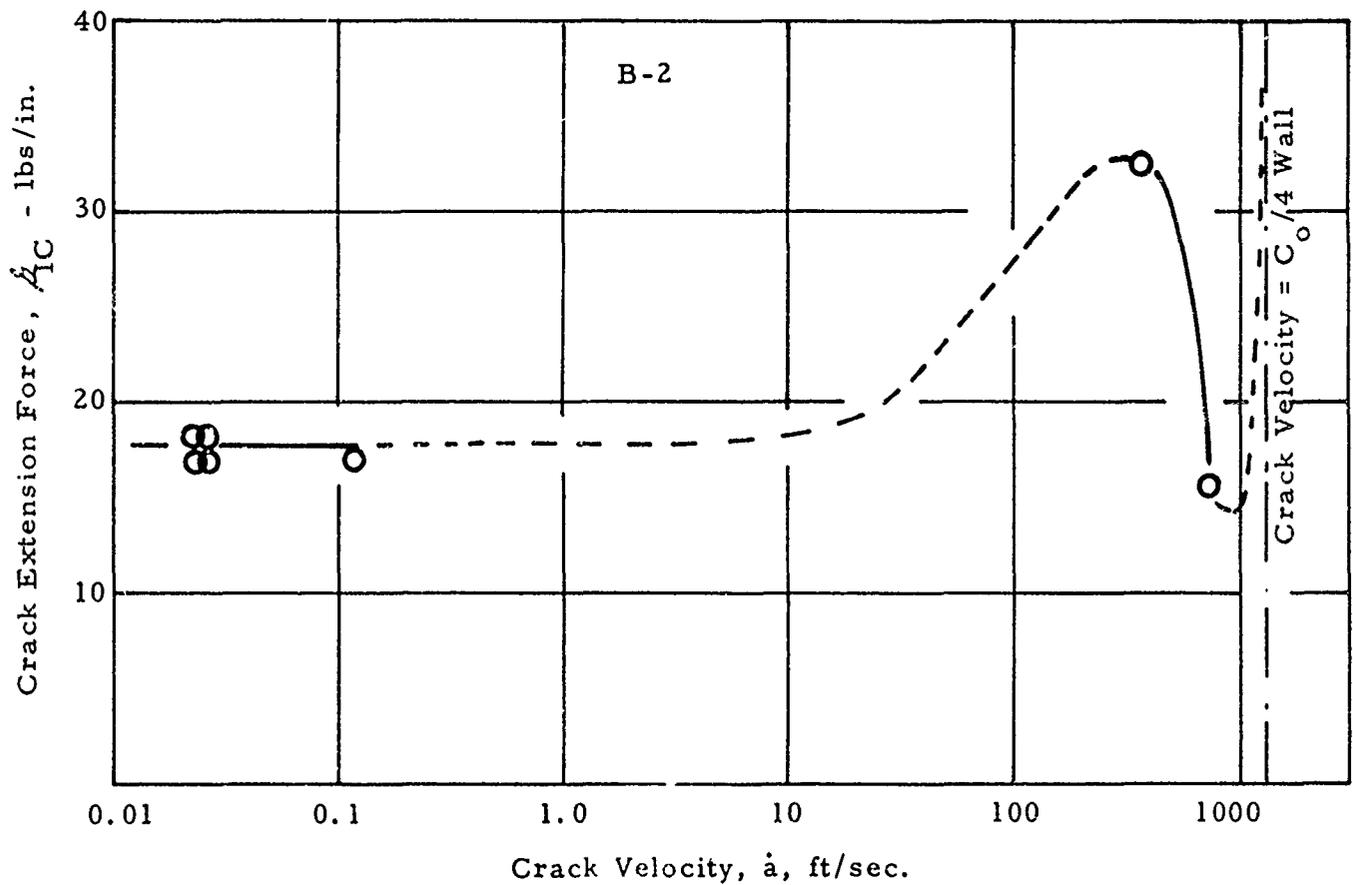Aluminum Adherends.   Highest speed obtained with m = 4 taper.
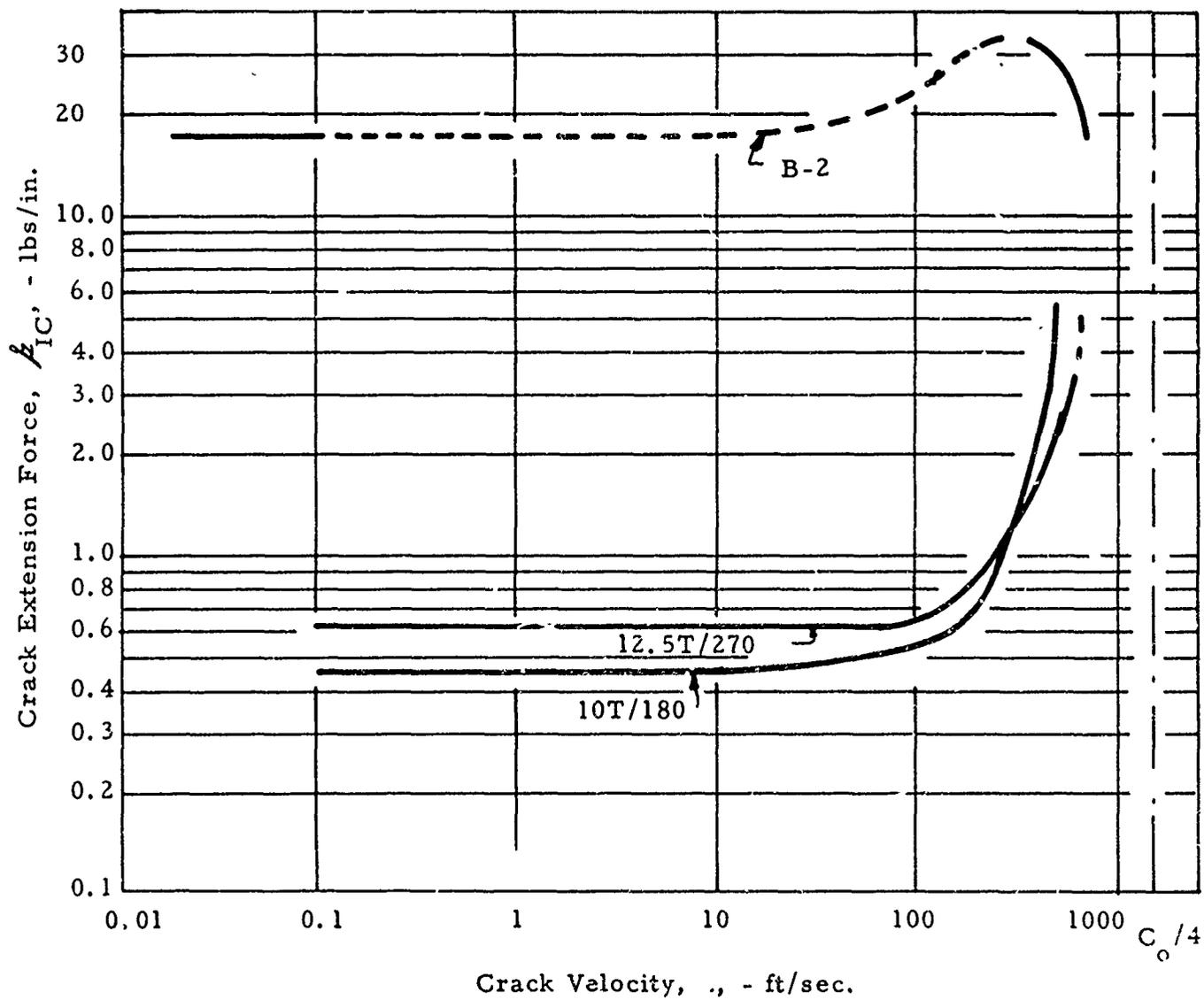(Data between 0.1 and 500 ft/sec. shown schematically.)

Fig. II-5   Toughness Comparison as a Function of Crack Speed for Three
Adhesive Systems.   Note that at high rates toughness of all
systems converge.

Section III

EFFECT OF PRIMER AND TEMPERATURE ON
STRESS CORROSION CRACKING

The most serious deficiency ' simple tw component adhesive systems is their inability to withstand c... .....ouerate crack-extension forces in the presence of liquid or vaporous water (1). The commercial adhesives, on the other hand, have excellent resistance to crack extension at room temperature in an aqueous environment (2). Indeed, the threshold value of crack-extension-force below which stress corrosion cracking (SCC) will not occur, $\mathcal{L}_{ISCC}$, cannot be measured since it does not lie below the value of plane strain fracture toughness, $\mathcal{L}_{IC}$. These water resistant commercial systems, however, always use primers, which, of course, raises the question of whether it is the adhesive, or primer that enables these systems to perform so well. For this reason, this study was undertaken to evaluate the influence of commercial primers in combination with a water sensitive adhesive.

The adhesives selected were DER 332 resin hardened with 12.5 PHR of TEPA, and post-cured at $270^{\circ}F$, identified as 332/12.5T/270 and the same resin with 10 PHR of hardener, post-cured at $180^{\circ}F$, and identified as 332/10T/180. The former system has an average $\mathcal{L}_{IC}$ of the order of 0.60 lbs/in. and a $\mathcal{L}_{ISCC}$ of 0.30 lbs/in. with cleaned but untreated aluminum adherends while for the latter $\mathcal{L}_{IC} = 0.30$ and $\mathcal{L}_{ISCC} = 0.08$ lbs/in. Earlier studies with this system had shown that cracking converted from center-of-bond, CoB, (typical of rapid cracking) to interface, IF, (typical of SCC) in less than four hours under load for unprimed adherend. With the use of a 0.5 w/o aqueous A1100 silane pre-

treatment of the adherends this "waiting time" was extended to about one month (about 720 hours). After this period, however, crack extension in the silane coated specimens was also reinitiated at the interface; and in spite of this long waiting period, the value of K_{ISCC} was not changed by the pretreatment. Crack extension, once begun, progressed as rapidly as when no pretreatment was used.

Obviously then, the primers used in pre-treating might act in either of two manners. On the one hand, they may simply delay the time required for the crack to switch from CoB to IF which appears to be the case for the silane, or they may actually completely prevent SCC. The latter is suggested by the data collected on certain commercial systems where even very long time exposures of CoB cracks to high loads in the presence of water did not lead to IF cracking. Consequently in the present study, one of the most popular of the commercial primers, BR 127, was used. To be sure that the primer was not simply delaying the time for conversion from CoB to IF, one specimen of a water susceptible adhesive was coated only on the back half. Hence, an IF crack could be easily generated in the front, uncoated section and extended into the coated one.

In addition, two commercial systems, one that uses a primer and one that does not were also evaluated for SCC at an elevated temperature. The one using a primer was a commercial rubber modified epoxy, referred to as B-2 in this program, and the other that does not consisted of Shell 826 resin hardened with 14.5 PHR of MPDA and post-cured at 340°F, referred to as 826/14.5M/340. Neither of these were subject to SCC in water (plus wetting agent) at room temperature at K_i values equal to or greater than average K_{IC} values.

TEST PROCEDURE

The aluminum adherends used for the specimens in this study were spray coated with 0.0002 to 0.0004 inches of BR 127. Coating thicknesses were determined as a function of passes for a particular spray gun on cured test coupons using an anvil micrometer protected from damaging the coating by use of a thin sheet of paper. In this range of thicknesses, the thickness of the coating could easily be found from a color comparison of the unknown with a set of such test coupons prepared to have a range of thicknesses. After spraying to obtain the required thickness, the coated parts were air dried at room temperature and then heated to 250°F for one hour to obtain a final cure. The coating, of course, was applied over the full surface to be bonded.

In one set of adherends used for evaluating the resistance of the primer to a crack running at the interface, only the rear half of the bond surface was coated. This was done by masking the front section of the cleaned adherends during the spraying process. Once coated and dried these adherends, like the others, were used to prepare a 5-mil bonded specimen, in this case using the 332/10T/180 adhesive.

The commercial B-2 adhesive used the primer recommended by the adhesive manufacturer, and the 826/14.5M/340 used no primer. The former had a 10 mil bond, produced by the recommended clamping pressure, and the latter had a 5-mil bond similar to all other poured adhesives.

RESULTS AND DISCUSSION

Two specimens of BR 127 primed 332/12.5T/270 were exposed to water containing a wetting agent (0.5 w/o Kodak Photoflo 200) starting with moderate values of applied crack-extension-force, $\mathscr{L}_i$. The values of $\mathscr{L}_{IC}$ and $\mathscr{L}_{IA}$ of these specimens, measured in the course of pre-cracking were at the upper limit of the scatter band for this adhesive system, 0.82 and 0.55 lbs/in., respectively. The value of $\mathscr{L}_i$ was increased, stepwise to values that were high even in relationship to these values of $\mathscr{L}_{IC}$. As shown in Table III-1, a total exposure of 101 and 111 days (approximately 2500 to 2600 hours) much of it at a $\mathscr{L}_i$ level of 0.76 lbs/in. produced no measurable crack growth. After this time, the specimens were removed from the loading fixture. The fact that cracking did not occur might have resulted because the time delay for conversion from CoB to IF was excessively long, or because SCC was actually prevented. To determine which of these is occurring, the two exposed specimens were subjected to corrosion fatigue in order to develop an IF fracture. This was not successful, however, and it was for this reason that a half-coated specimen was used. This specimen (332/10T/180) was precracked in the unprimed region to obtain the $\mathscr{L}_{IC}$ value and a normal CoB fracture. The specimen was then subjected to SCC in the presence of water. The value of $\mathscr{L}_i$ was such that crack initiation and growth began in the area of the unprimed interface and extended toward the primed bond surface at the rear of the specimen. During this SCC crack extension, standard crack growth rate vs. $\mathscr{L}_i$ data were obtained. The SCC behavior of this unprimed area of the specimen showed the low $\mathscr{L}_{ISCC}$ value, i. e. 0.09 lbs/in. typical of this adhesive. As the crack driven at relatively fast rates ($\dot{a}$) by a high $\mathscr{L}_i$ value approached the primed bond area there was a marked slow down in rate. Increases in $\mathscr{L}_i$ did not result in an

increased rate.   The crack was completely arrested at a $\mathcal{K}_i$ value of 0.22 lbs/in. after it had transversed the 0.80 inch transition area between primed and unprimed bond surfaces.   Further time at this high $\mathcal{K}_i$ level did not cause crack extension although the specimen was maintained under load for approximately 1000 hours.   The standard $\mathcal{K}_i$ vs. $\underline{\dot{a}}$ curves, Fig. III-1, is shown with the results of the crack extension study through the transition region to the primed area.

The use of the commercial primer BR127 appears to prevent SCC not simply by delaying the time required for the crack to convert from CoB to IF as appears to be the case for silane, but even cracks extending at the interface are unable to propagate.   Examination by the scanning electron microscope of SCC surfaces suggest that crack extension in an aggressive environment occurs by corrosion.   Hence the primers not only provide a high level of adhesion at the primer-metal (oxide) and primer-adhesive interface, but also prevent corrosion of the metal surface at room temperature.

The two adhesives that had been previously shown to resist SCC at room temperautre, i.e. B-2 and 826/14.5M/340 were also exposed to water at elevated temperatures.   Although these tests have not been sufficiently completed to define $\mathcal{K}_{ISCC}$ as a function of temperature, preliminary data collected to date suggest that at $130^{\circ}F$, a commonly used test temperature, the rate at which SCC occurs is appreciable.   The expected $\mathcal{K}_{ISCC}$ value as a function of elevated temperature water exposure is plotted in Figs. III-2 and III-3.   From these curves, and the IF fracture surfaces observed after testing, it is seen that SCC in water will occur in these systems at only moderately elevated temperatures.

REFERENCES

1.   E. J. Ripling, S. Mostovoy and C. Bersch, "Stress Corrosion Cracking of Adhesive Joints", J. Adhesion, 1971, Vol. 3, p. 145.

2.   Sheldon Mostovoy and E. J. Ripling, Final Report, "Factors Controlling the Strength of Composite Structures", Contract No. N00019-70-C-0137, for the period March 1, 1970 through February 28, 1971.

Table III-1

LOADING HISTORY IN WATER OF 332/12.5T/270 ADHESIVE BOND
(5 MIL THICKNESS) BETWEEN BR 127 PRIMED ADHERENDS

Specimen #4124

Applied μ_i	Time at Load Δ_t	Total Time Under test t
lbs./in	hours	hours
0.15	24	24
0.17	24	48
0.35	4	52
0.37	20	72
0.40	4	76
0.43	20	96
0.46	24	130
0.48	5	135
0.50	19	144
0.52	168	312
0.54	312	624
0.76	1800	2424*

Specimen #4123

Applied μ_i	Time at Load Δ_t	Total Time Under test t
lbs./in.	hours	hours
0.16	4	4
0.17	4	8
0.35	2	10
0.37	6	16
0.40	5	21
0.43	3	24
0.46	8	32
0.48	2	34
0.50	6	40
0.52	8	48
0.54	80	128
0.60	72	200
0.70	168	368
0.76	2280	2648*

*No crack growth, removed from fixture

Crack Growth Rate, $\dot{a}$, mils/min.

50
40
30
20

10.0
8.0
6.0

4.0
3.0

2.0

1.0
0.8
0.6

0.4
0.3

0.2

0.1

<0.01

O Unprimed Crack Growth
□ Primed-unprimed Transition Region Crack Growth
△ Primed Crack Growth

Crack at start of transition region

$\mathcal{G}_{ISCC}$ (unprimed) $\cong 0.09$ lbs/in.

Crack slows through transition region

P

P

0.8"

Unprimed area

Fully primed

Transition region

$\mathcal{G}_{IC}$

Crack arrested by fully primed area

0   0.10   0.20   0.30   0.40   0.50   0.60

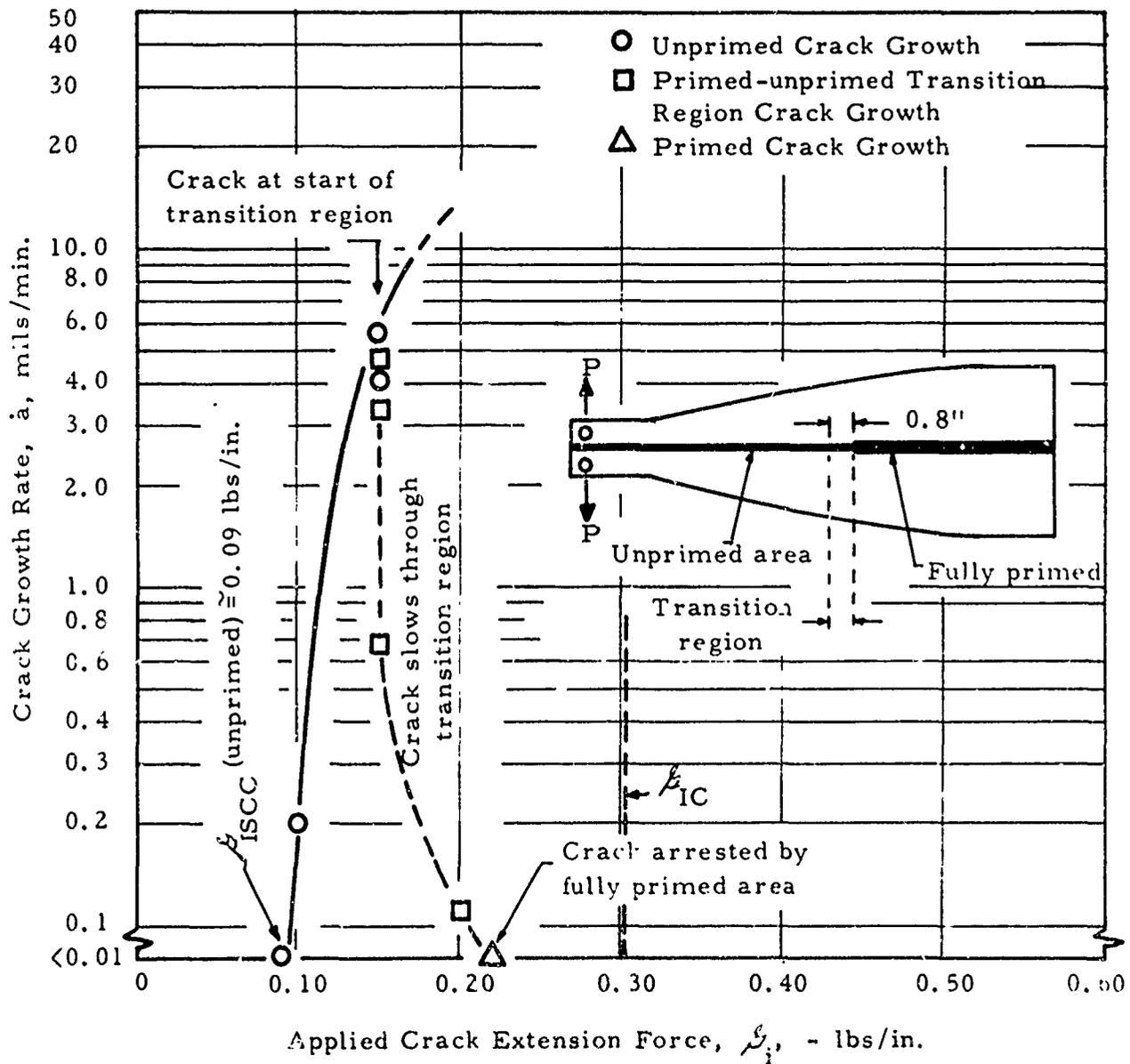Applied Crack Extension Force, $\mathcal{G}_i$, - lbs/in.

Fig. III-1 Behavior of Partially Primed Aluminum Adherend Adhesive Specimen when Exposed to Liquid Water Stress Corrosion Cracking (adhesive material 332/10T/180; 5 mil bond, m = 90 in.$^{-1}$ adherends).
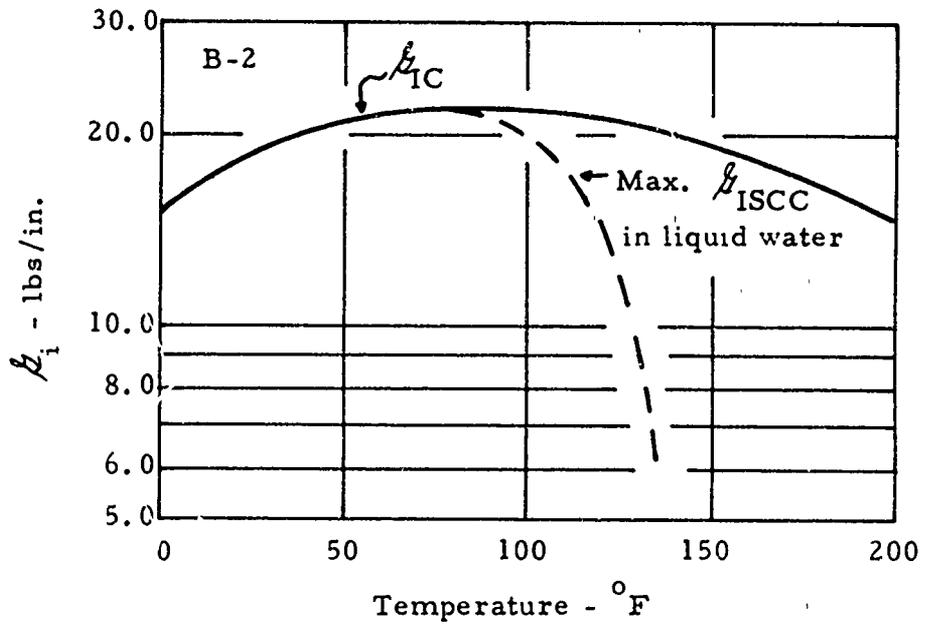
Fig. III-2   Stress Corrosion Cracking of B-2 Adhesive in Liquid Water as a Function of Test Temperature.
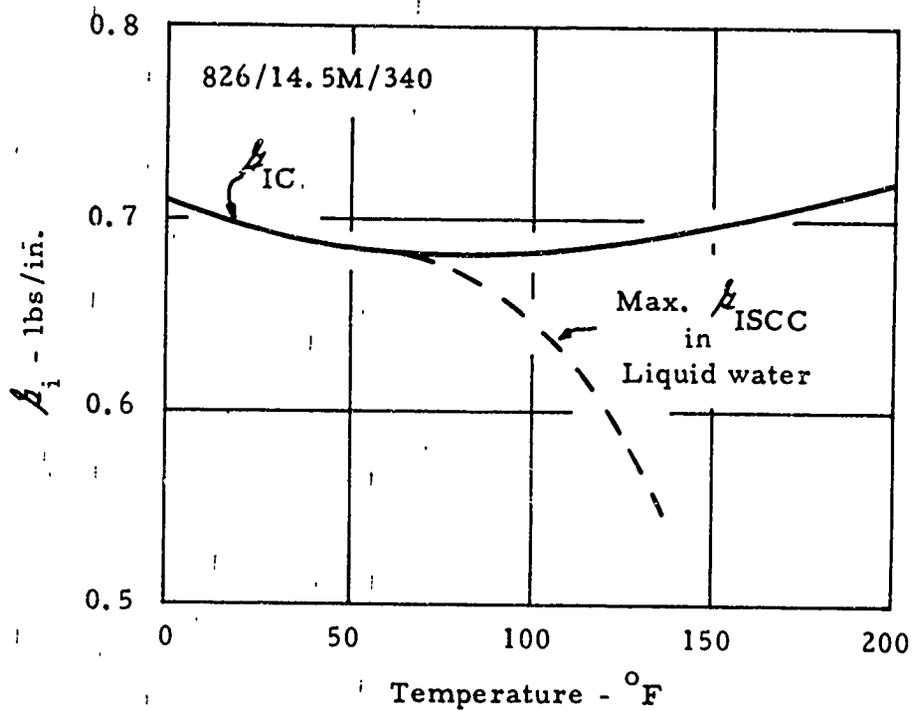
Fig. III-3   Stress Corrosion Cracking of 826/14.5M/340
Adhesive in Liquid Water as a Function of
Test Temperature.

Section IV

FATIGUE AND CORROSION FATIGUE

This laboratory has described the fracturing characteristics of
adhesive joints under a wide range of service conditions. Loading rates
have been studied from those fast enough to cause the crack to approach
its limiting velocity to those slow rates that result from sustained loads.
Test temperatures have varied from that of liquid nitrogen to those in
excess of the post-cure temperature; and environments have varied from
essentially dry through increasing relative humidities to liquid water.
During this past year this regime of service variable was extended to in-
clude fatigue and corrosion fatigue. The data collected under this condi-
tion to date are quite limited, however, and must still be considered pre-
liminary.

MATERIALS AND PROCEDURE

Three adhesive systems were selected for this study, none of
which exhibited stress corrosion cracking in water at room temperature.
The first of these was Shell 826 hardened with 14.5 PHR of MPDA, post-
cured at $340^\circ F$ and used without a primer, the second was DER 332,
hardened with 12.5 PHR of TEPA, post-cured at $270^\circ F$, primed with
BR 127, and the other was a commercial rubber modified, filled, "B"
staged film adhesive used with the primer recommended by the manu-
facturer. These are identified as 826/14.5M/340, 332/12/5T/270 and B-2,
respectively, throughout this report.

Specimen preparation, including treatment of adherends, and pre-cracking to form the initial center-of-bond (CoB) cracks were identical to that previously described. In all cases fatiguing was started with CoB pre-cracked specimens so that one might expect some delay time if the fatigue crack were to extend at the interface, IF.

The fatigue cycle was selected to approximate one now being used at the Naval Research Laboratory, and on which a large amount of data has been collected on GRP composites. One complete cycle requires 90 second: 27 second rise time, 60 second hold at maximum load, and 3 second fall to a load slightly above zero, with no hold-time at the low load. Load is never dropped completely to zero to maintain loading alignment during the test.

Fatigue testing was done in an ambient atmosphere (i. e. 40-60 percent RH) and for corrosion fatigue, a small dam was built around the joint to hold the distilled water plus wetting agent (0. 5 w/o Kodak Photoflo 200) during cycling. All testing was done at room temperature.

RESULTS AND DISCUSSION

826/14.5M/340

Three specimens of this adhesive system were subjected to the above described fatigue cycle in an ambient humidity. The maximum applied $\mathcal{G}_i$ value and test results on each of these is shown in Table IV-I. It is apparent that with repeated loading, an applied $\mathcal{G}_i$ (0. 41 lbs/in.) of the order of $\mathcal{G}_{IC}$, is sufficient to cause relatively fast sub-critical cracking (10. 2 mils/cycle or 6. 8 mils/min.) even though this adhesive system was not sensitive to stress corrosion cracking in water under the

action of a static load. Further, the crack extended at the interface in the 50 percent relative humidity (R.H.) exposure. For well made adhesive joints, interface cracking in the past has only been found when the test environment was aggressive; hence, one might expect that 50 percent R.H. is corrosive for this system in the presence of a repeated load although it is not for a static load.

332/12.5/270

One specimen of this adhesive using the BR 127 primer ($3 \pm 1 \times 10^{-4}$ inch thick) was fatigue loaded in 50 percent R.H. in a manner identical to that described above for the 826/14.5M/340 adhesive.

The $\ell_{i(max)}$ first applied in 50 percent R.H. was somewhat less than $\ell_{IC}/2$, Fig. IV-1. When it was found that this did not cause cracking, $\ell_{i(max)}$ was increased in approximately 0.1 lbs/in. steps to just below ℓ_{IC}. Roughly 500 cycles were used per step. Since cracking did not occur, even after cycling at this high value of ℓ_i, it was assumed that the previous long time, low ℓ_i exposure might have toughened the joint. Consequently, after obtaining $\ell_{i(max)} = 0.7$ lbs/in., and more than 2000 total cycles, the specimen was recracked in the static test machine to form a new center-of-bond crack. Cyclic loading was continued, again at $\ell_i = 0.7$ lbs/in. for 3000-4000 more cycles. Since crack extension still did not occur, $\ell_{i(max)}$ was increased well above ℓ_{IC} for this specimen (see Fig. IV-1) again with no crack extension. After approximately 500 cycles at $\ell_{i(max)} = 0.9$ lbs/in., the specimen was unloaded and exposed to liquid water at about 2/3 the ℓ_{IC} value. After no cracking was observed for several days, the specimen was again uploaded to ℓ_i approximately equal to ℓ_{IC}. The cycling was continued for more than ten days with no indication of crack extension.

B-2

One specimen of this commercial adhesive was subjected to the same type of loading pattern as that described above, again in 50 percent R.H. starting with $\mathcal{G}_{i(max)} < \mathcal{G}_{IC}/2$. When it was found that cracks extended at a controlled rate in this adhesive system, quantitative cracking rate data were collected as follows: The specimen was cycled at a relatively high value of $\mathcal{G}_{i(max)}$ until the crack began to move. Crack motion during cycling was measured using compliance; a load displacement chart was made for the particular cycle, and compared with the first cycle. After 50 to 100 cycles, the cracking rate was seen to stabilize, and this stable value was plotted in Fig. IV-2. The cyclic load was then decreased and the process repeated. Once $\mathcal{G}_{i(max)}$ for no growth was obtained, which took more than one week, the load was again increased to allow the crack to continue moving. The threshold value of $\mathcal{G}_{i(max)}$ to be consistent with the fracture mechanics literature is indicated as $\Delta\mathcal{G}_{th_{50}}$ where the subscript 50 indicated 50 percent R.H. The value for $\Delta\mathcal{G}_{th_{50}}$ for this adhesive is about 4.5 lbs/in. which is about 1/3 to $\frac{1}{2}$ the value of $\mathcal{G}_{IC}$. Nevertheless, this value is substantially above $\mathcal{G}_{IC}$ for all the simple (essentially two component) systems, and many of the commercial systems as well.

After completing the exposure in 50 percent R.H., the specimen was further fatigued in liquid water. Surprisingly, the fatigue threshold, $\Delta\mathcal{G}_{th}$, in liquid water exposure is higher than for 50 percent R.H. Thus, water is seen to toughen the system in a fatigue environment.

After final fracture, the fracture surface was examined and it was found to be center-of-bond (CoB). This fact coupled with the rate and $\Delta\mathcal{G}_{th}$ data for liquid water and 50 percent R.H. confirms that crack

extension in this case occurs by fatigue alone. The toughening effect of water has been seen before in the increasing load tests of the simple adhesive systems. It was found that water added to the crack tip after precracking increased the toughness over the levels obtained in cracking in ambient humidity (50 percent R.H.).

CONCLUSIONS

Since only a modest amount of data has been collected to date on fatigue and corrosion fatigue, conclusions regarding this type of load application on joints must be considered tentative. Nevertheless, some behavior patterns are proposed:

1. Since the 826/14.5M/340 adhesive exhibited IF fracturing at $\mathscr{k}_{i(max)}$ values less than $\mathscr{k}_{IC}$ in 50 percent R.H. when subjected to static loads, it may be concluded that fatigue is more damaging to the interface than static loads.

2. The 332/12.5T/270 adhesive using the BR 127 primer could not be fatigue cracked in either 50 percent R.H. or liquid water, even at values of $\mathscr{k}_{i(max)}$ about 25 percent higher than $\mathscr{k}_{IC}$. This might suggest that if the interface is well protected some minimum value of $\mathscr{k}_{i(max)}$ is necessary for fatigue cracking, and $\mathscr{k}_{i(max)}$ may lie above $\mathscr{k}_{IC}$ for simple two component systems.

3. For adhesive systems in which the interface is well protected and which have a high value of $\mathscr{k}_{IC}$ allowing for slow cracking in fatigue (e.g. B-2) water

may toughen the crack so that fatigue resistance in a dry environment is poorer than it is in a wet (water) environment. Of course, this would not be applicable at elevated temperature since it does not appear possible to prevent IF cracking in warm (say $130^{\circ}F$) water.

Table IV-1

FATIGUE BEHAVIOR OF 826/14.5M/340 ADHESIVE[*]

Specimen	$\mathcal{G}_{i(max)}$	Incubation Time	Cracking Rate After Incubation		Fracture Morphology
			da/dN	$da/d\epsilon$	
	lb/in.	cycles	mils/cycle	mils/min.	
A	0.61	44	159	105	Interface
B	0.61	43	35.3	23.2	$\frac{1}{2}$ interface $\frac{1}{2}$ herringbone (CoB)
C	0.41	500	10.2	6.8	Interface

Notes:

1. Cycle - 27 sec. rise, 60 sec. hold, 3 sec. fall reload with no hold time at 0 load.

2. Environment - Ambient (40-60% R.H.)

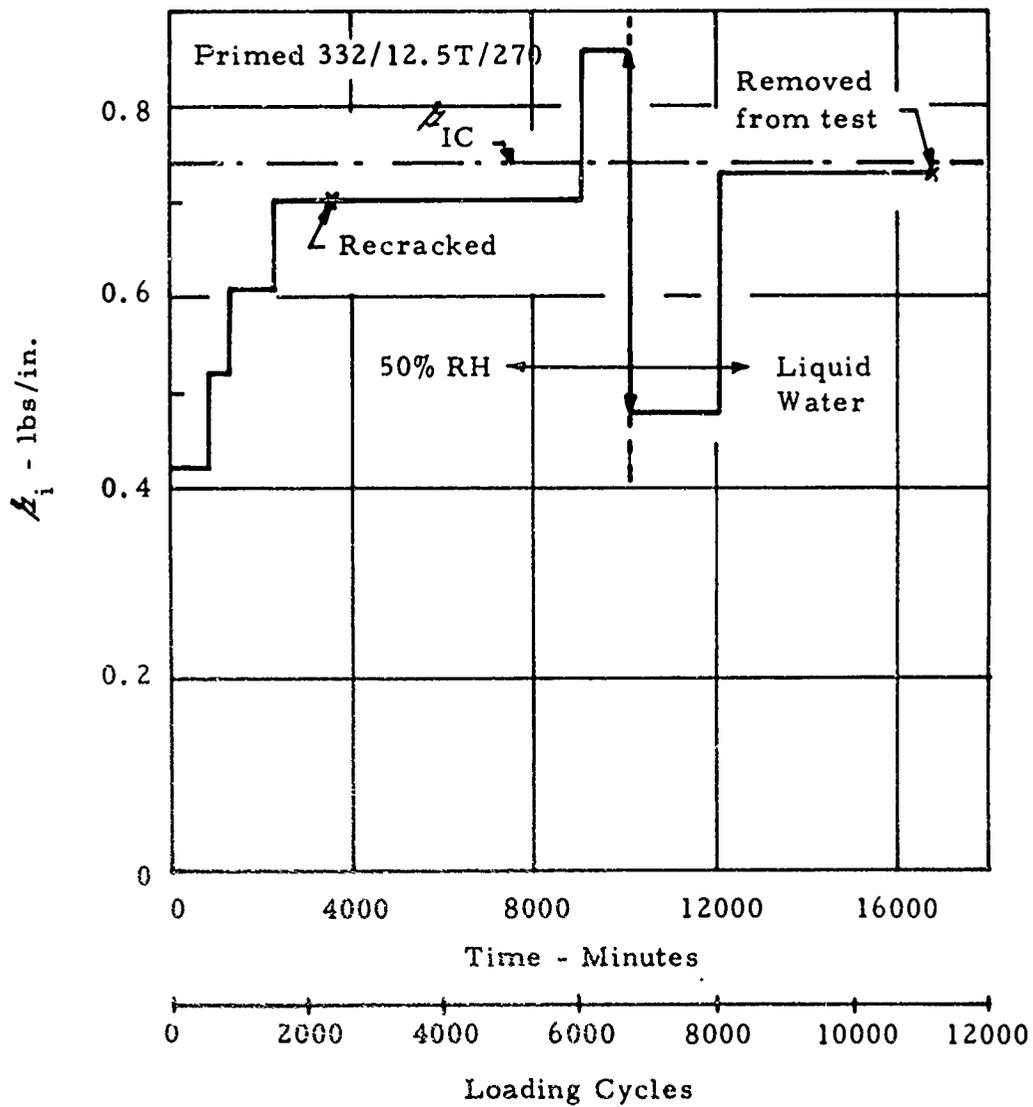3. $\mathcal{G}_{IC}$ range - 0.75-0.90 lbs/in. $\mathcal{G}_{Ia}$ = 0.66 lbs/in.

Fig. IV-1   Corrosion Fatigue History in Liquid Water and 50% RH
for 332/12.5/270 Adhesive.   BR 127 Primed Aluminum
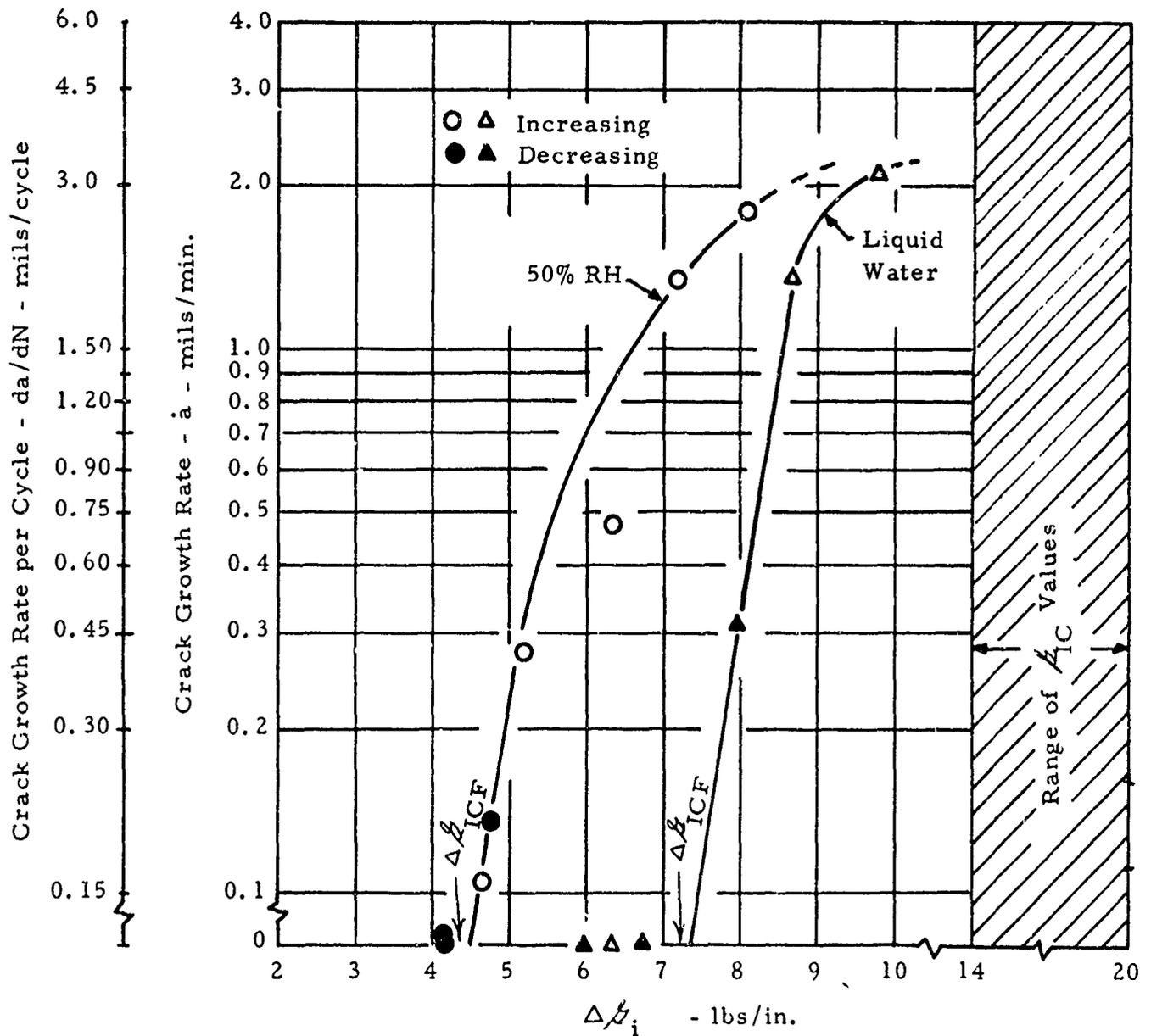Adherends, 5 mil bond.   (No cracking observed during
exposure.)

Fig. IV-2   Effect of Corrosion Fatigue Cycling at Room Temperature
on Cracking rate of   B - 2   Structural Adhesive for
Liquid Water and 50% RH Environment.   The 90 second
Fatigue Cycle Consists of a 27 second Loading, a 60 second
Hold at Maximum Load and a 3 second Unloading to Near
Zero Load.