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Quarterly Progress Report #2

For the period May 17, 1964 to August 17, 1964

on

FACTORS CONTROLLING THE STRENGTH OF COMPOSITE BODIES  
(INTERPHASE FRACTURING OF COMPOSITE BODIES)

Contract No. NOW 64-0414-c

for

BUREAU OF NAVAL WEAPONS  
Washington 25, D. C.

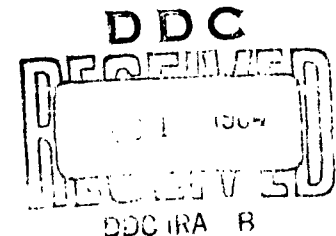
by

E. J. Ripling and S. Mostovoy

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## ABSTRACT

The effect of mechanical and chemical processing variables on the fracture toughness of adhesive joints was investigated. Two adhesive systems were used, viz., Budd Photostress Type A and Dow 332 plus TEPA. Most of the studies were made on aluminum adherends although some data were collected on steel and glass adherends as well.

Fracture toughness was markedly influenced by the chemical variables, which included epoxy system, ratio of epoxy to hardener, and post-cure temperature. Budd A was not only the tougher of the two at the stoichiometric composition (14.7 PHR) but it also underwent a three-fold increase in toughness when the hardener content was increased from stoichiometric to 20 PHR. The toughness of DER 332-TEPA also increased with hardener content, but at a lower rate. Further, the toughness of the latter epoxy also increased with post-cure temperatures up to 210<sup>o</sup>F after which the toughness decreased.

These changes in toughness were not apparent in smooth tensile properties of bulk epoxy samples. Fracture toughness data on the bulk epoxy has not been collected as yet.

The mechanical variables, bond thickness, adherend smoothness, and cracking rate exerted only a small influence on toughness. Very thin and very thick bonds exhibited an increased scatter band that is associated with a change in fracture type.



## INTRODUCTION

As was noted in the previous report (1) the epoxy system was changed from Budd Photostress A to Dow 332 plus TEPA hardener at the beginning of this contract period. Over the present report period, the chemical and mechanical process variables in producing joints with these two materials were studied. The testing program involved a study of the bulk smooth tensile properties of the Dow 332-TEPA system as a function of hardener content and post-cured temperature. The influence of these same two chemical processing variables on joint toughness for both systems were also evaluated. The mechanical variables, including the effect of joint thickness, surface finish and crack velocity with a variety of adherends were also studied. The oscilloscope and ripple pattern technique described in the previous report has been used to determine  $\dot{L}_{IC}$  for a running crack.

Work on glass during the previous contract indicated that an increase in surface roughness improved joint toughness. Tests have also been conducted during this report period on steel and aluminum specimens of varying roughness to quantitatively evaluate this effect. Motion pictures were taken of a moving crack with the glass adherends in order to collect data on the crack front length.

Work on glass adherends is continuing to evaluate surface finishes and treatments commonly used in filament reinforced plastics. In order to collect the most useful data, plates of Corning 1720 glass, similar to the "E" glass used in fiberglass, is being obtained in a limited supply from W. Bascom, at NRL.

A new specimen having a constant  $\partial(1/M)/\partial a$  developed for strain rate work, has been successfully tested and enables a determination of  $\dot{L}_{IC}$  knowing only the load. Knowledge of instantaneous or fixed interval crack lengths will still be developed in order to calculate crack velocities.



## PROCEDURES AND RESULTS

The variables that control the strength of a joint subjected to an opening mode fracture can be classified as chemical or mechanical. The former includes adherend and adhesive composition and curing procedures, and the latter, bond thickness, adherend surface finish, and cracking rate. These two classes of variables are discussed separately in the following.

### Chemical Variables

Tensile test data on solid epoxy collected for two post-cure temperatures and varying epoxy formulations are shown in Fig. 1. While the number of data points are limited, trends in the various mechanical properties are apparent. For either post-cure temperature, hardener in excess of the stoichiometric amount (14.7 PHR) has a plasticizing effect, i.e. total and plastic elongation\* ( $\underline{e}_t$  and  $\underline{e}_p$ ) increase while Young's Modulus (E) decrease appreciably, and the tensile yield ( $F_{ty}$ ) and ultimate tensile strength ( $F_{tu}$ ) drop slightly or not at all. There is a difference in level for the two post-cure temperatures, which includes a lowering of modulus and yield strength accompanied by an increased elongation for the higher temperature cure.

As hardener percentage is lowered below stoichiometric, there is little change in any of the properties other than  $\underline{e}$  for either post-cure temperature, but again a change in property level for the two post-cure

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\* Since the value of elongation is thought to depend on the flaw in the sample, these curves are drawn to represent the expected top of the scatter band.



temperatures. One would expect a drop in properties at very low hardener content (e.g.  $\leq 10$  PHR), and specimens are now being prepared to examine both the very high and very low hardener systems.

Solid epoxy  $G_{Ic}$  toughness specimens are also being prepared to evaluate hardener content and post-cure temperature, but the problem of exotherm at high hardener levels requires that lower initial curing temperatures or cycles be found to approximate the small tensile specimens and the bonded specimens where exotherm was less of a problem because of the reduced epoxy volume. Thus the entire curve especially at high hardener levels will probably not be defined with solid epoxy toughness bars.

To compare "as-bonded toughness" properties with bulk properties, two series of  $G_{Ic}$  specimens were prepared; one to evaluate amount of hardener, the other to evaluate the effect of post-cure temperature. These data are shown in Fig. 2.

Earlier reports (2) (3) indicated that the toughness of adhesive joints depended on the shape of the load-deflection curve, i.e. "flat" fractures generally had a higher toughness than "peaked" ones. To classify a  $P-\delta$  curve as one or the other of these, its shape after the crack started running is compared with the  $P-\delta$  curve for constant  $G_{Ic}$ . Those that lie above the constant  $G_{Ic}$  curve are classified as flat, and those that lie on or below it are denoted peaked. Data in 2a were plotted for peaked failures only, even though they represented less than 1/3 of the total.

The amount of flat vs peaked behavior and the amount of "interfacial" fracturing (defined as the area, in percent, of the crack that travelled at or very near the interface as compared with the total fracture area) were plotted for the increasing hardener series in Fig. 3.

When Budd A and Dow 332 were compared at their stoichiometric compositions (14.7 PHR) (Fig. 2a) toughness values for the two were within

20 percent of each other. However, when the hardener was increased to 20 PHR the Budd material was 300 percent tougher than the Dow epoxy, even though the latter was about 50 percent higher than it was at the stoichiometric composition. The rate of change of toughness with hardener at the higher hardener level is extremely steep for the former.

When hardener is decreased from stoichiometric to 12 PHR, the toughness of the DER-332 remained essentially constant; below 12 PHR it decreased.

This difference between the two epoxies when used as joints is not evident from their bulk tensile properties; at a TEPA content of 20 PHR, DER-332 had an  $E = .455 \times 10^6$  psi,  $F_{tu} = 11.3$  ksi,  $F_{ty} = 5.5$  ksi,  $e_t = 9.86\%$ , and  $e_p = 2.38\%$ , while for Budd A (4),  $E = .92 \times 10^6$  psi,  $F_{tu} = 9$  ksi,  $F_{ty} = 4$  ksi,  $e_t = 3.65\%$ , and  $e_p = 1.6\%$ . Note that the toughness of the Budd epoxy exceeded that of the Dow by 300 percent while the relative tensile properties are reversed.

For the stoichiometric composition, the DER 332 system (Fig. 2b) shows a continually increasing toughness with increasing post-cure temperature up to about  $210^\circ\text{F}$  after which there is a general loss of toughness associated with interfacial fracturing and some deterioration of the epoxy as evidenced by discoloration and "powdering" at the bond surface.

In a study of surface work of fracture on several solid epoxy systems, McGarry (5) presented data from which  $\frac{K_{Ic}}{E}$  could be calculated for Shell 828 epoxy with 15 PHR TEPA in which the post-cure temperature varied from ambient to  $300^\circ\text{F}$ . The toughness continuously increased over this temperature range, and other epoxy systems showed similar results. While the trends for the bulk epoxy and joint material are similar, the bonded data goes through a peak at a much lower temperature (and toughness) than the solid epoxy system.



Additional specimens are being prepared to more fully define both the effect of hardener content and post-curing temperature.

Fig. 3 shows the dependence of type of fracture on hardener content. As the amount of hardener is increased, a larger percentage of the fracture takes place near the interface. (Additionally the  $P-\delta$  curves show a predominantly "flat" behavior.) Although "flat" fractures were eliminated in Fig. 2 because of the supposed larger crack front length, many "peaked" fractures were seen where fracturing did occur at the interface with higher toughness than those that fractured at some distance from the interface. If it is assumed that the hardener migrates to the metal adherends, the joint would have a composition gradient consisting of "plasticized" interphase layers, and a hard center. Fractures that are restricted to the interface would consequently have a high toughness.

#### Mechanical Variables

Figs. 4, 5 and 6 are plots of the effect on  $K_{IC}$  of bond thickness, surface finish and crack velocity, respectively. These are considered to be mechanical effects although surface finish, thin bonds and fracture near or at the interface are likely to be related to chemical phenomena.

In Fig. 4, the effect of bond thickness is shown for Budd and Dow epoxy systems of two hardener concentrations using aluminum adherends. Over the range of investigation, 1 to 500 mils, the effect of a bond thickness change on toughness was negligible except for a considerable scatter of both very thick and very thin bonds. The apparent higher toughness at both extremes of bond thickness, especially for the Budd material, is partially understood by the difficulty in defining crack front length,  $b$ , in the expression for toughness. In moderately thin bonds the crack front was equal to



the specimen width, while in both thick and thin bonds (including solid epoxy  $\frac{L}{l_c}$  specimen) the crack front was much longer than this width in many cases. This was noted earlier with aluminum adherends when photoelastic patterns were seen to extend well ahead of the crack front and with glass adherends where direct visual observation of crack width could be made. At thin bonds, the problem is compounded by a tendency of the crack to run at the interface and give no indication of crack front length to specimen width ratio.

A plot of surface finish versus toughness in Fig. 5 shows that the adherend material is apparently much more important in determining adhesive toughness than the smoothness of the adherend surface. In the earlier work, using glass adherends and Budd A epoxy, an increase in surface roughness lead to an increase of toughness to that value obtained with aluminum adherends. Joints prepared using smooth glass did not appear to be well bonded and could easily be removed with a penknife. As roughness was increased, the epoxy did appear to be more difficult to remove. To date only smooth glass adherends have been used with DER 332, however, the toughness values obtained were about half that for aluminum adherends. This ratio is comparable with the Budd A epoxy for smooth glass. Steel adherends with the same finish as smooth glass, i.e. 1 - 3  $\mu$ in., were also found not to bond well with 332 and TEPA, but, unlike the smooth glass, toughness values were quite high. This is misleading, as in the case of bond thickness, possibly because of the elongated crack front and interfacial fracturing conditions which predominate in steel adherends. Increasing roughness did not cause an improvement in joints with steel adherends.

Fig. 6, the strain rate or crack velocity effect, while containing only a modest amount of data, indicates that over the probable limits of standard machine capability little rate effect will be noted. With the low loading rate



running crack velocities have been obtained using ripple pattern and oscilloscope techniques. For a 2-1/2 orders of magnitude velocity increase, toughness is decreased by about 20 percent. This is assumed to be the largest effect because a driven crack would most likely require a greater  $K_{Ic}$  for propagation at this rate. The plot also includes head velocity data at an intermediate crack length for driving a crack at the velocities plotted. For a Fig. 7 type specimen, head velocity for a given crack velocity is constant over all crack lengths and equal to 5.22"/min. for a 1 ft./sec. crack and aluminum adherends. As is noted, the fastest driven crack in an Instron tester will travel at about 15 ft./sec. If higher driven velocities are necessary to determine the predicted minimum, either a machine faster than the Instron or deeper beam specimens will have to be used.

With adherends of uniform cross section, the velocity of a driven crack continuously decreases when  $\dot{\gamma}$  is held constant. By tapering the test specimen, as shown in Fig. 7,  $d(1/M)/da$  was made constant over the bonded length so that toughness was proportional to  $P^2$ . Specimens with this shape simplify the strain rate study.

#### Glass Adherends

One of the aims of the program is to establish fracture toughness data on glass adherends with a view toward applications in glass reinforced systems. A limited amount of water white plate glass adherends has been obtained, a few of which have already been used for the motion picture work to define elongated crack width effects. Work using these adherends as well as Corning 1720 plate supplied in limited quantity, courtesy of W. Bascom at NRL, will be continued during subsequent report periods to determine the effects on toughness of various surface finishes and treatments among those in common use in fiberglass manufacture.



## CONCLUSIONS

The effect on fracture toughness of the mechanical and chemical variables involved in producing adhesive joints are being studied. Since only a limited amount of data has been collected to date, the following conclusions must be considered as tentative.

1. Although differences in bulk smooth tensile properties were comparatively minor between Budd A and Dow 332 plus TEPA systems, fracture toughness differed by a factor of two or more. Further, changes in tensile properties for a given epoxy system as a function of hardener content or post-cure temperature gave little indication of the toughness changes in a cracked bonded structure. A comparison of bulk epoxy toughness with as-bonded toughness indicates that these two properties may be similarly influenced by chemical variables.

2. The chemical variables, i. e. epoxy and adherend compositions, and post-cure temperature, have a far greater influence on toughness than the mechanical variables which include joint thickness, adherend surface finish, and crack velocity.

3. The large scatter in toughness of Budd A epoxy when used as a joint which had previously been reported (2) may have resulted from the steep slope of the composition-toughness curve at 20 PHR.

4. The positive slope of the toughness vs hardener content curve results not only from the increased plasticizing effect of the excess hardener but also from a greater tendency for "interphase" fractures. The latter may result from a migration of the hardener toward the adherends.

5. It was possible to develop a constant  $\partial(1/M)/\partial a$  specimen by properly tapering the adherends. With this sample shape, toughness is proportional only to the load squared so that controlling strain rates even



over long cracking lengths is readily accomplished. Crack rate on a fine scale is readily measured by an observation of ripple patterns produced by an oscillation technique described in Ref. (1).

6. The difficulty of defining fracture toughness in joints that results from different fracturing behaviors, i. e. peaked vs flat has not been completely resolved although the higher toughness of flat fractures appears to be related to chemical and surface effects that cause abnormally long crack front lengths. The complexity resulting from two fracture modes was found in joints made with smooth steel adherends; although the measured toughness values were high, the epoxy could be readily stripped from the steel surface. The misleading high measured toughness values probably result from excessively long crack fronts.

#### FUTURE WORK

Once a more complete picture of the effect of chemical variables is known, the influence of mechanical variables will be more easily understood.

Strain rate effects are presently under investigation using the tapered, constant compliance  $[\partial(1/M)/\partial a = \text{constant}]$  specimens shown in Fig. 7. The problem of exact determination of crack length to obtain  $K_{IC}$  will be eliminated. Only in those cases where the crack velocity outruns the loading device will crack lengths need to be determined directly. For the most part, only load and cracking rate need be known as the crack moves. The applicability of this specimen has been shown for a slow speed toughness test with results comparable to the standard double beam specimen.

Solid epoxy tensile and toughness bars are being prepared to complete Figs. 1 and 2.



The results of the toughness vs PHR hardener curves for the solid material should indicate whether numerical conversion to  $\mathcal{G}_{Ic}$  is possible for the Orowan surface work values obtained by McGarry (5). In addition the conjecture of a stratified or migrated hardener, used to explain much of the interface fracture toughness data, and the tendency for interfacial fracturing at high PHR systems will be checked.

Solid epoxy toughness specimens will be prepared both as the standard double beam and as in the Fig. 7 to get a comparison between the two types.

Quantitative measurements of the influence of  $\mathcal{G}_{IIc}$  on  $\mathcal{G}_{Ic}$  (and vice versa) have not been made to this time because of the difficulty in resolving loads and deflection in the bonded specimens. However, early quantitative results indicated that  $\mathcal{G}_{IIc}$  was at least one order of magnitude higher than  $\mathcal{G}_{Ic}$  and that even a small  $\mathcal{G}_{II}$  component substantially increased  $\mathcal{G}_{Ic}$  (2). Work during the latter part of this contract period will be done using separate  $\mathcal{G}_I$  and  $\mathcal{G}_{II}$  loading fixtures to avoid the problem of the resolving of loads and deflection. A plot of  $\mathcal{G}_{Ic}$  vs  $\mathcal{G}_{IIc}$  will be developed by separately preloading in one direction and loading to fracture in the other direction.

Early work using Budd A epoxy on glass adherends showed that smooth (i.e. 1 - 3  $\mu$ in.) glass adherends had a fracture toughness value  $\mathcal{G}_{Ic}$ , about 1/2 that of rough bond surface (i.e. 150  $\mu$ in.) glass or aluminum adherends. This same toughness ratio has been observed for smooth glass versus aluminum adherends (i.e., .16 vs .32  $\mu$ in<sup>2</sup>) for the Dow 332 TEPA system. The work using "rough" glass will be continued during this period and motion pictures will be taken to determine the initiation and behavior of a running crack. Attempts will also be made to induce the crack to run at some distances from the interface using a teflon tape crack starter and a thicker bond.

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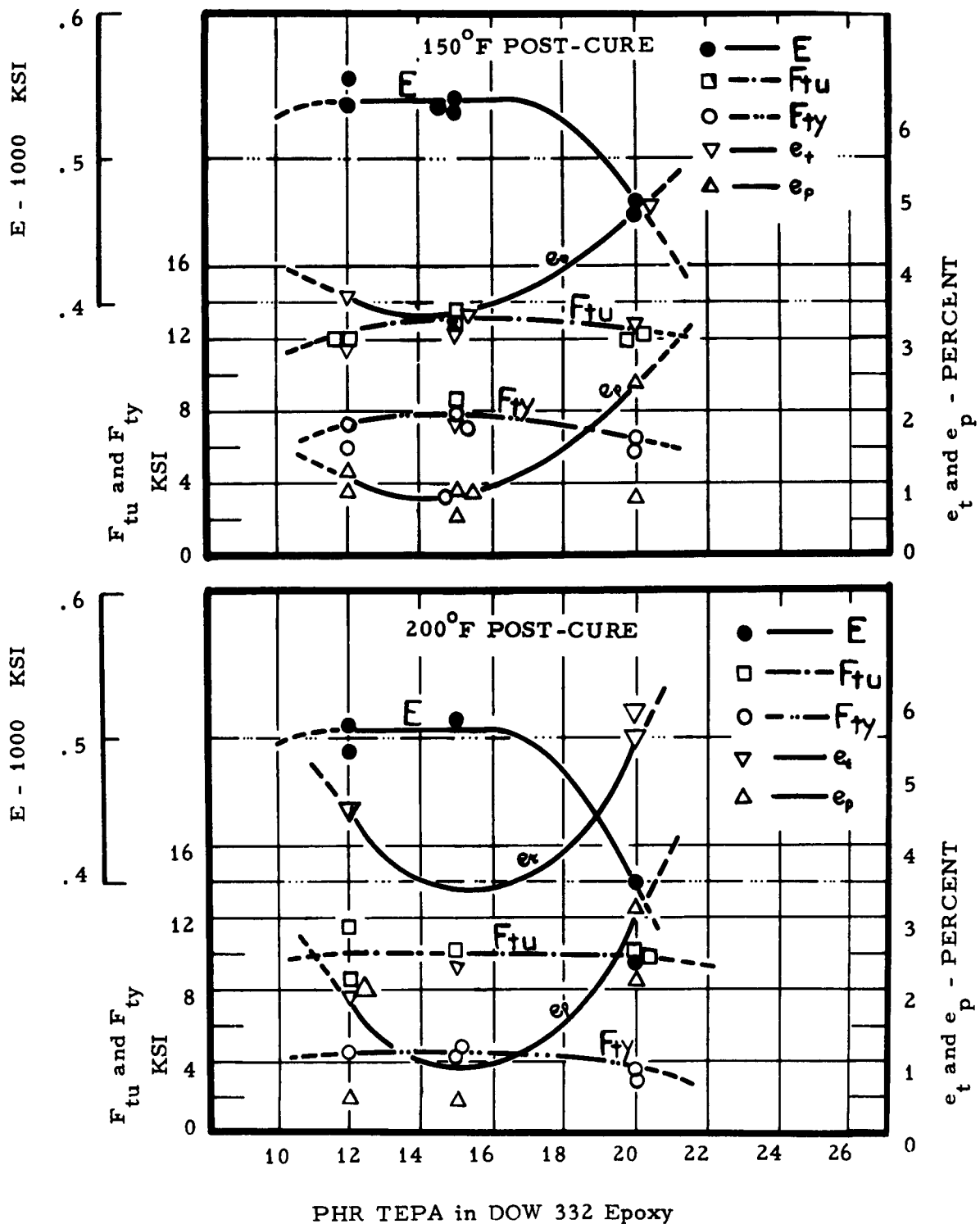
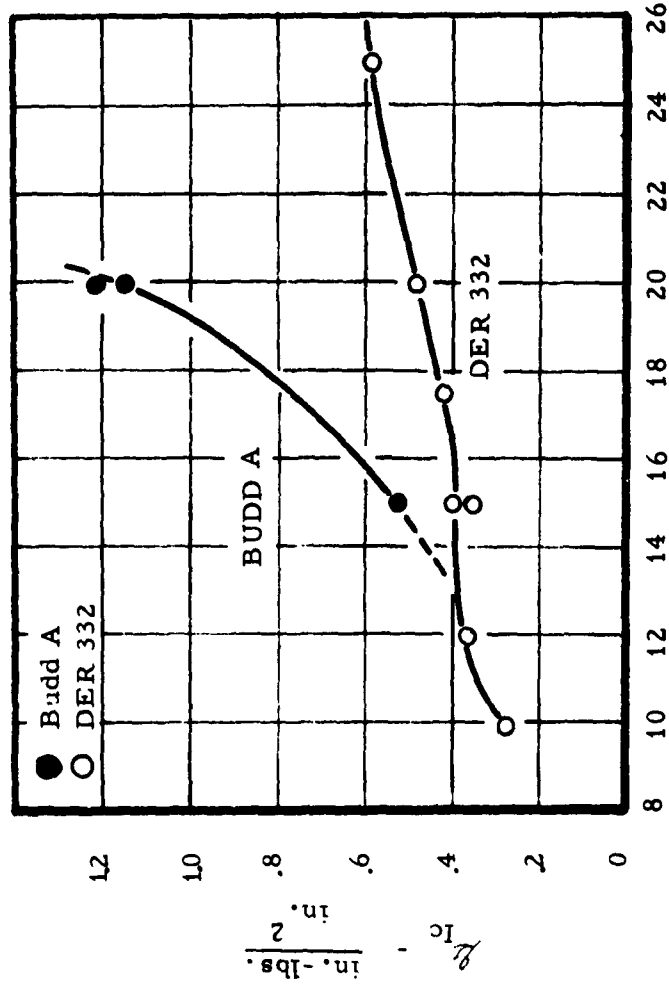
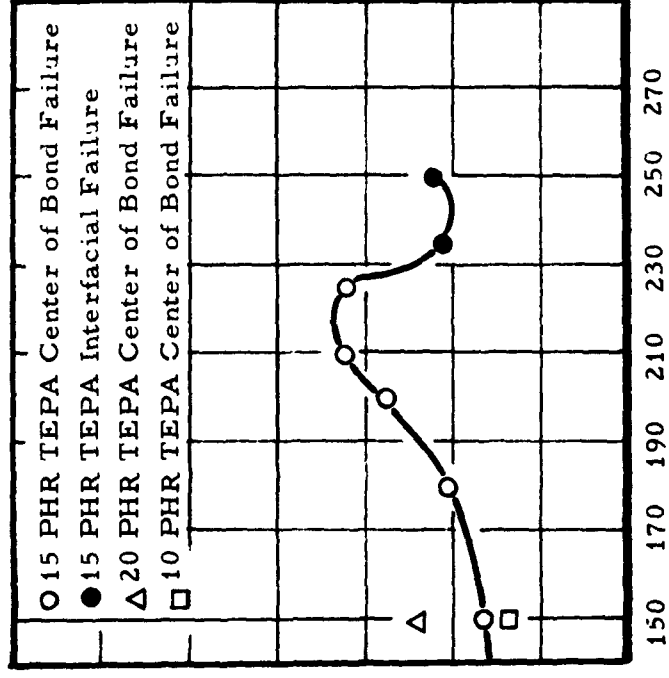


Fig. 1 TENSILE TEST DATA OF SOLID EPOXY SPECIMENS NOMINALLY 1/8 x 3/8" ON A 2" GAGE LENGTH FOR TWO POST-CURE TEMPERATURES, 150 and 200°F. VARIABLES PLOTTED ARE YOUNG'S MODULUS (E), ULTIMATE TENSILE STRENGTH (F<sub>tu</sub>), TENSILE YIELD (F<sub>ty</sub>), TOTAL AND PLASTIC ELONGATION (e<sub>t</sub> and e<sub>p</sub>).



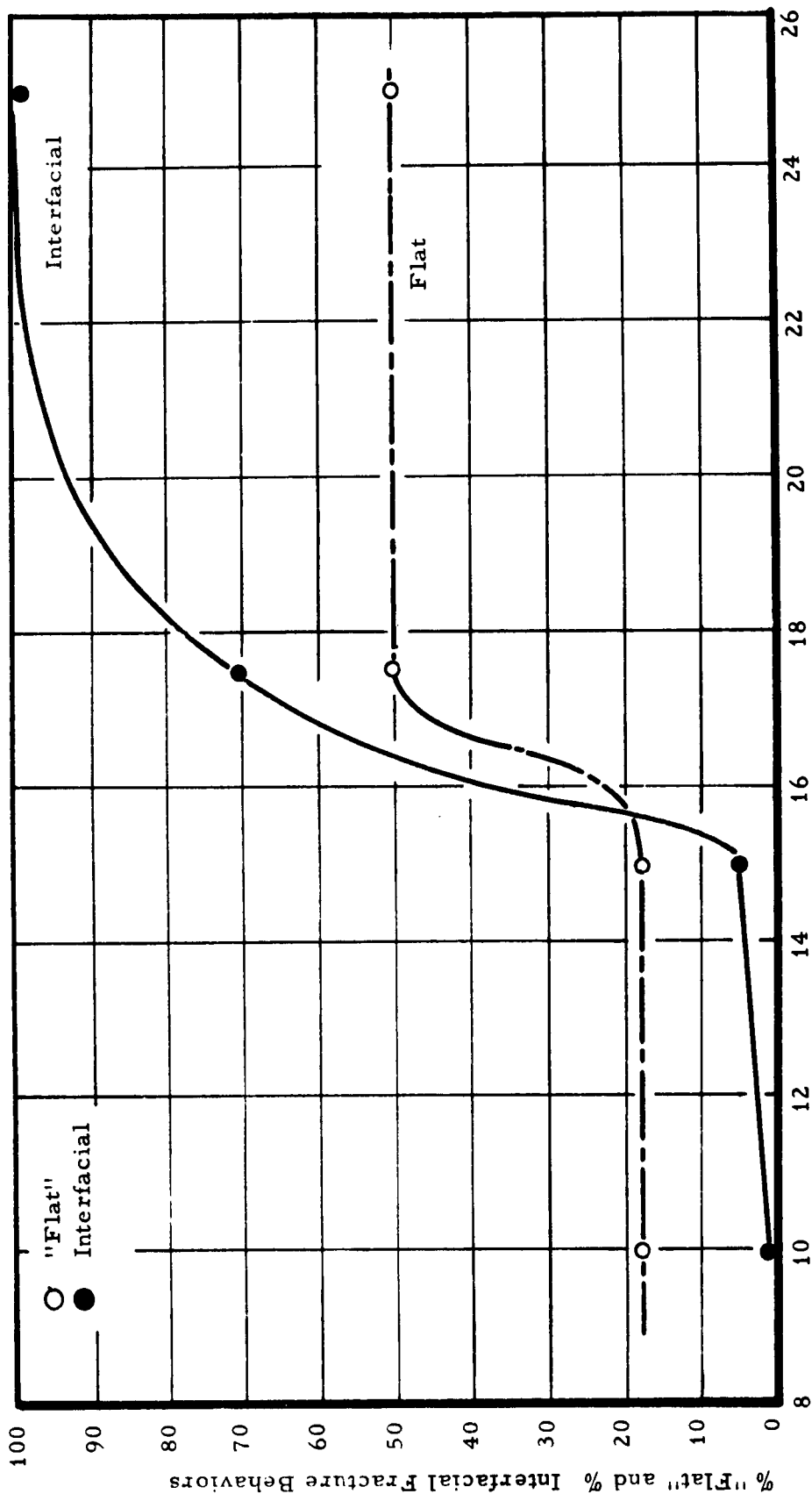
PHR TEPA or Budd A Hardener  
in Dow 332 or Budd A Epoxy

Fig. 2a  $L_{10}$  SPECIMENS PREPARED WITH 5 MIL BONDS USING 1/2" ALUMINUM TO EVALUATE EFFECT OF PHR HARDENER (150°F Post Cure; "Peaked" values only;  $t_c = 5$  to 15 sec.)



Post Cure Temperature - °F  
(5 hours at Temp.)

Fig. 2b  $L_{10}$  SPECIMENS PREPARED WITH 5 MIL BONDS OF DER 332 USING 1/2" ALUMINUM TO EVALUATE THE EFFECT OF POST CURE TEMPERATURES. ("Peaked" values only;  $t_c = 5$  to 15 sec.)



PHR TEPA Hardener in Dow 332 Epoxy

Fig. 3  $L_{16}$  SPECIMENS EXAMINED AS TO FRACTURE APPEARANCE AND SHAPE OF P- $\delta$  CURVES OBTAINED DURING TESTING (on 5 Mil Bond, 1/2" Aluminum; 150° F Post Cure;  $t_c = 5$  to 15 sec.)

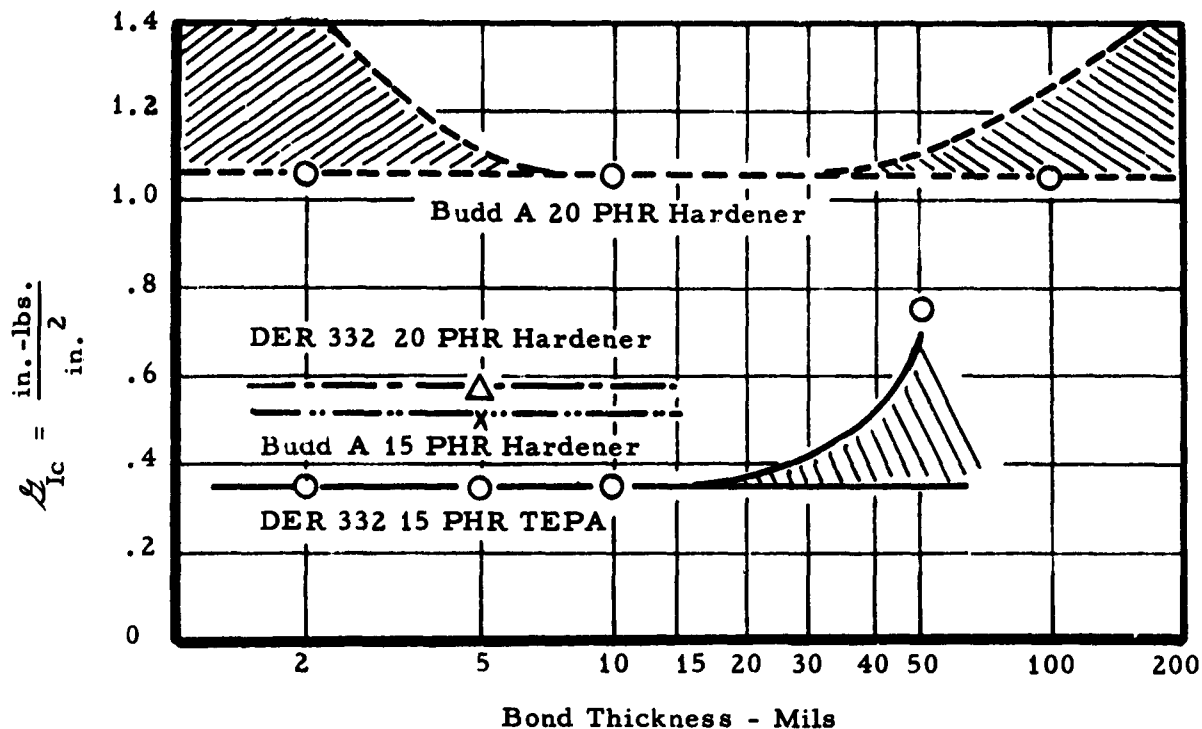


Fig. 4 EFFECT OF BOND THICKNESS ON  $L_{Ic}$  FOR ALUMINUM ADHERENDS 1 in. TALL USING BUDD A AND DOW 332 EPOXIES WITH VARYING PERCENTAGES OF HARDENER. (Surface finish = 10 $\mu$ in;  $t_c$  = 5 to 15 sec.)

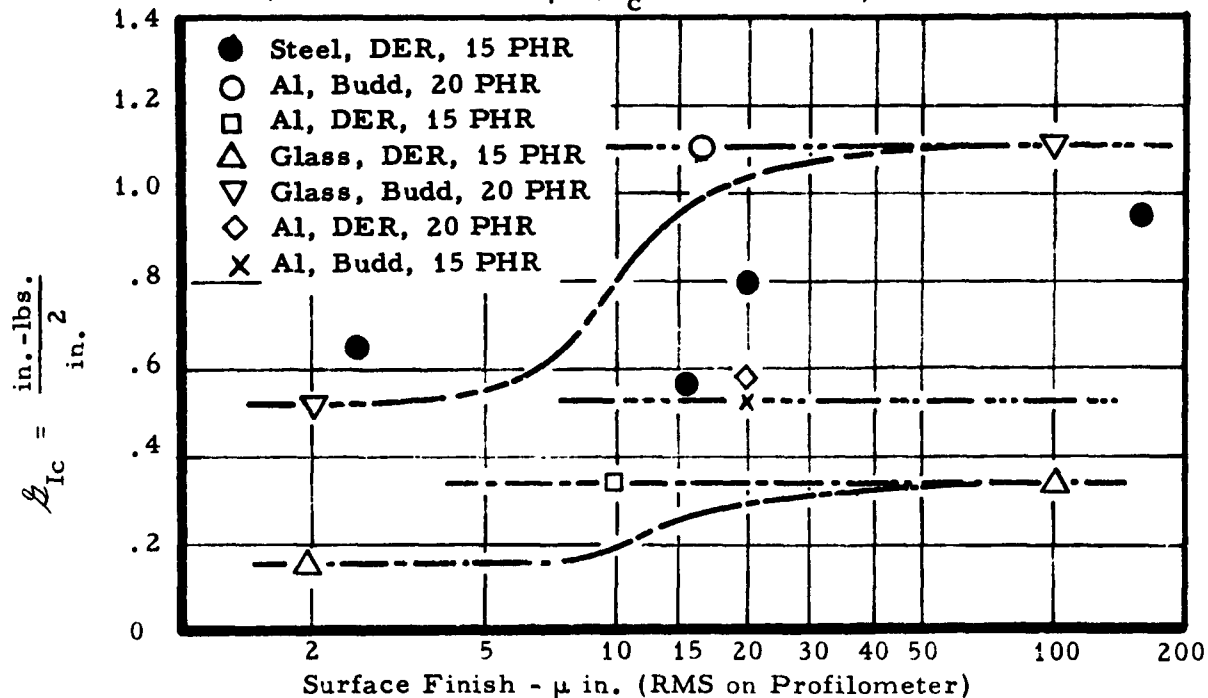


Fig. 5 EFFECT OF SURFACE FINISH ON  $L_{Ic}$  USING STEEL AND ALUMINUM ADHERENDS WITH 5 MIL BONDS OF DOW 332 EPOXY 15 PHR TEPA AND 150 $^{\circ}$ F POST CURE ( $t_c$  = 5 to 15 sec.)

Head Velocity to Maintain Corresponding Crack Velocity  
in 1" Tall Aluminum Adherends for a 5" Long Crack.

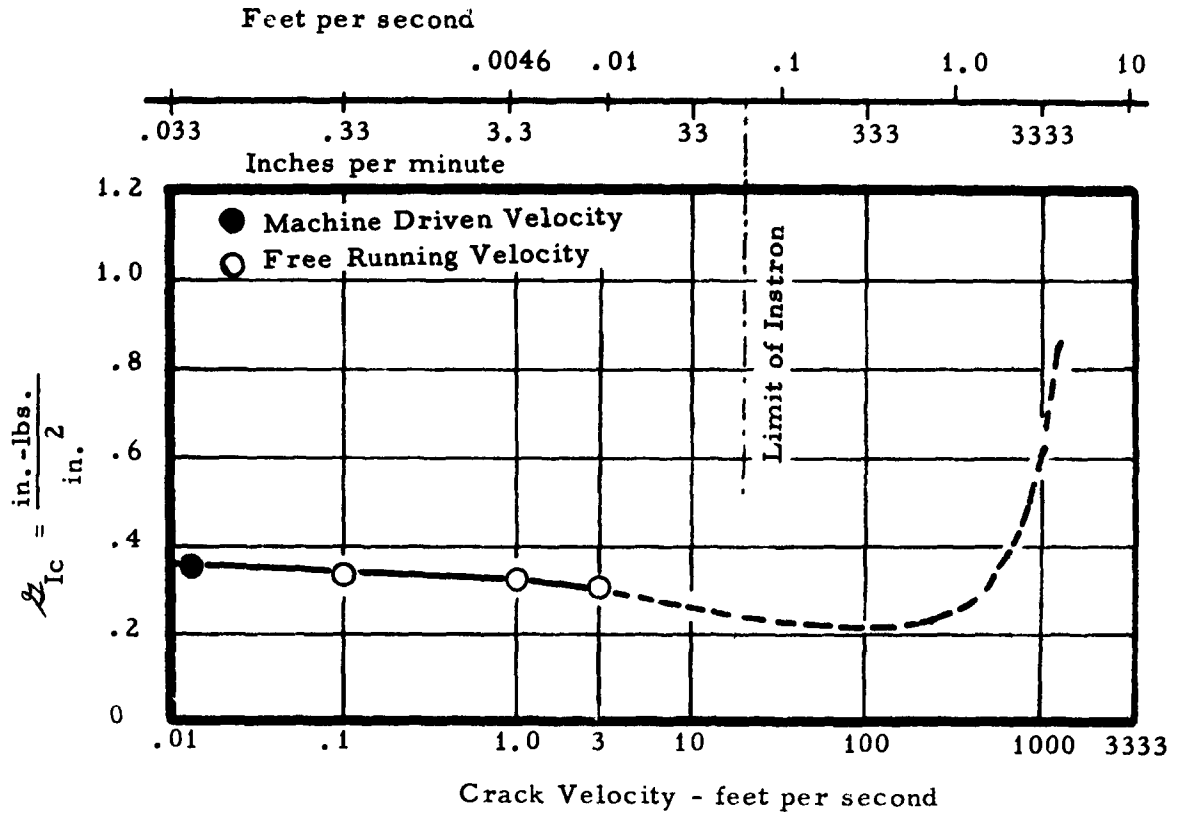


Fig. 6 EFFECT ON  $G_{Ic}$  OF CRACK VELOCITY FOR 1/2" WIDE 1" TALL ALUMINUM ADHERENDS BONDED WITH DOW 332 EPOXY AND 15 PHR TEPA HARDENER (5 MIL BONDS) VELOCITIES OF RUNNING CRACKS DETERMINED USING OSCILLOSCOPE AND OSCILLATOR MARKED FRACTURE SURFACE TECHNIQUES. NOTE HEAD VELOCITIES NECESSARY TO DRIVE CRACKS AT VELOCITIES OF INTEREST.

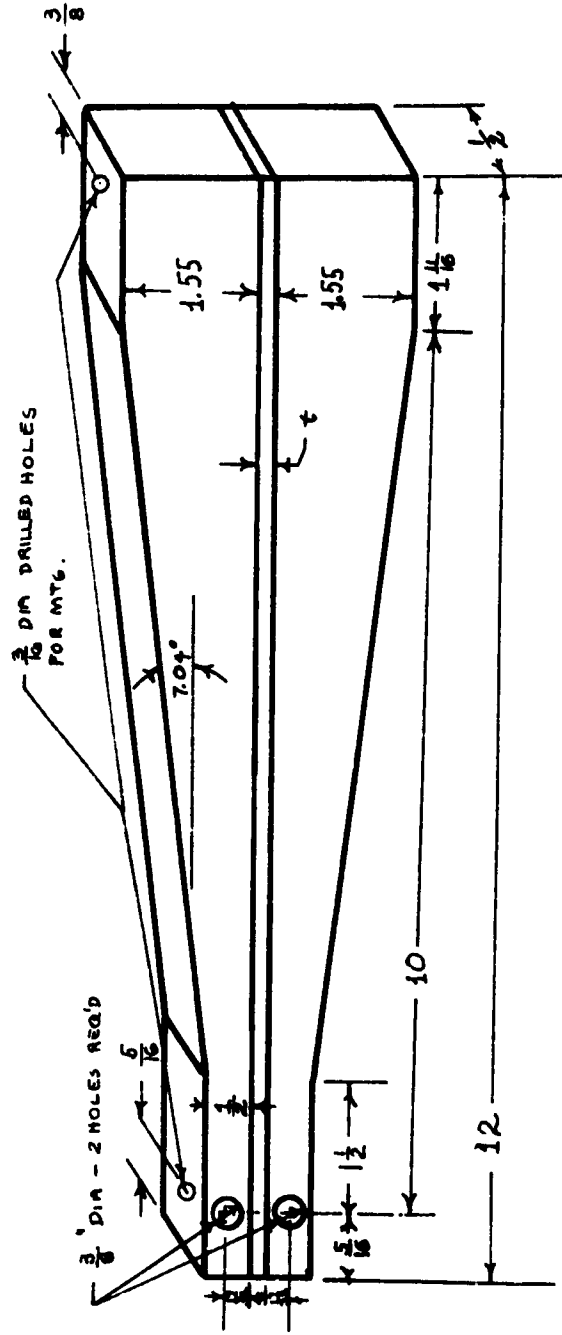


Fig. 7 CONSTANT COMPLIANCE ( $d(1/M)/da$ ) TAPERED  $A_{1c}$   
 ADHESIVE BONDED SPECIMEN (For Aluminum  
 $d(1/M)/da = 142 \times 10^{-6}$  lbs. .  $t$  is the bond thickness)



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Attn: Mr. Wm. McKay

Douglas Aircraft Corp.  
Missile and Space Systems Division  
3000 Ocean Park Blvd.  
Santa Monica, California  
Attn: Mr. J. H. Cunningham

Goodyear Aerospace Corp.  
1210 Massillon Road  
Akron 15, Ohio  
Attn: Mr. R. A. Berkeley, Dept. 481

Avco Corporation  
Undersea Projects Directorate  
201 Lowell Street  
Wilmington, Massachusetts

Union Carbide Plastics Company  
P. C. Box 670  
River Road  
Bound Brook, New Jersey 08805  
Attn: Mr. A. S. Burhans

Minnesota Mining and Mfg. Co.  
Reinforced Plastics Division  
2501 Hudson Road  
St. Paul 10, Minnesota

Monsanto Research Corp.  
Everett, Massachusetts  
Attn: Dr. Lucius Gilman

Hercules Research Center  
Wilmington 99, Delaware  
Attn: Mr. J. T. Paul

Professor Fred McGarry  
Massachusetts Institute of Technology  
Cambridge 39, Massachusetts

General Electric Company  
Space Sciences Lab  
P. C. Box 8555  
Philadelphia 1, Pa.  
Attn: Mr. B. Walter Rosen

The RAND Corporation  
Santa Monica, California  
Attn: Dr. George A. Hoffman

P. R. Mallory and Company, Inc.  
Technical Services Lab  
Indianapolis 6, Indiana

Bell Telephone Laboratories, Inc.  
Murray Hill, New Jersey  
Attn: Mr. R. Sabia

Professor James L. Lubkin  
Department of Civil Engineering  
Michigan State University  
East Lansing, Michigan

Mr. J. Marian  
University of California  
Forest Products Laboratory  
1301 South 46th Street  
Richmond, California