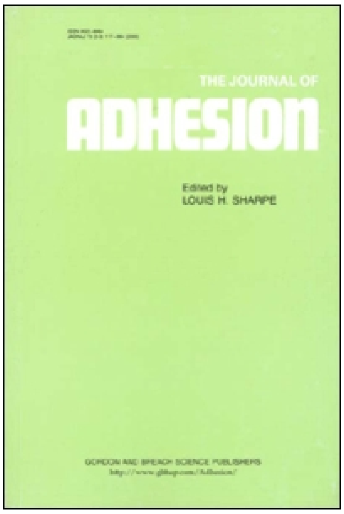


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# Mechanical Characterization of Adhesive Layer *in-situ* and as Bulk Material

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# Mechanical Characterization of Adhesive Layer *in-situ* and as Bulk Material

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An extensive test series was conducted on bulk and *in-situ* adhesive specimens with a view to characterizing their mechanical properties under different loading modes and states of stress.

It was found that a good correlation exists between the *in-situ* and the bulk properties of shear yield strength and elastic modulus derived from torsion tests. The properties derived from uniaxial testing of the bulk adhesive were related to those of an *in-situ* adhesive layer in shear by a combined stress law which follows a modified Von Mises failure criterion. It was thus concluded that the basic elastic and strength characteristics of the *in-situ* adhesive under a compound state of stress may be evaluated through simple tests on the bulk material in uniaxial tension and compression.

## INTRODUCTION

One of the main handicaps in the structural application of adhesively bonded joints is the complexity of their stress and failure analysis. This complexity, and the absence of an acceptable standard design- and testing-methodology for mechanical characterization, have caused the bonded system to be regarded as sensitive and unpredictable. At the engineering level this makes for impaired confidence in these joints and is probably one of the reasons for the current preference for conventional mechanical means of fastening—rivets and bolts—in structural elements made of both metallic and composite materials.

In the light of these doubts it is advisable to examine the feasibility of treating the adhesive not as a distinct phase but as one more regular structural layer within the multimaterial lamination sequence. Naturally, allowance must be made for its specific mechanical characteristics compared with the common FRP laminae or metallic adherends. By such an approach physical and mechanical premises may be applied and advantage taken of available theories, testing tools, and methodology used in the characterization of any multilayer structural system.

The following premises were postulated in the present work:

1) The basic mechanical behaviour of an *in-situ* bonded adhesive layer is similar to its corresponding bulk adhesive reference.

2) A simplified elasto-plastic model (defined by its initial elastic moduli and its yield stress plateau) suffices for the approximate prediction of the mechanical behaviour of the bonded adhesive layer under static loading.†

3) Failure of a properly surface-treated bonded joint is cohesive and initiates within the adhesive layer.

4) With regard to its environmental behaviour in time, an adhesive obeys laws similar to those which dictate the time-dependent hygrothermal behaviour of ductile polymers.†

The research project, part of which is reported here, was undertaken with a view to examining the hypothesis under which the adhesive layer is to be treated as another lamina within the composite laminate. This objective comprises three stages:

1) Establishment of the relationship between bulk and *in-situ* characteristics.

2) Study of the behaviour under compound stress, *versus* that under simple uniaxial loading.

3) Investigation of the effects of strain rate, temperature, and humidity on the mechanical characteristics of the adhesive layer.

Further objectives were: verification of the first three premises, and a new methodology for the mechanical characterization and the design of structural adhesive bonding.

## EXPERIMENTAL AND THEORETICAL BACKGROUND

Mechanical characterization of structural adhesives currently involves a wide variety of specimen types and loading procedures[1] whose limitations preclude derivation of adequate design allowables and largely restrict the evaluation to a comparative study of surface treatments, environmental effects, etc. In most

† Premises 2 and 4 are limited to rubber-modified adhesive systems (such as FM 73), which are characterized by high ductility.

cases the main drawback is that the compound state of stress within the adhesive layer is nonuniform and complicated: in particular the high stress concentrations close to the ends of the bonded joint are difficult to detect experimentally and to analyze—especially in terms of failure criteria—due to the singularity of this region in terms of material and geometrical discontinuity. (As regards strains and displacements, they are strongly dependent on adherend constraints and also very difficult to measure, mainly due to the small and nonuniform thickness of the adhesive layer.) One exception is the Napkin Ring specimen, in which the state of stress is almost uniform pure shear, and several investigators[2–10] have proposed instrumentations and techniques for studying, by this means, the effect of thickness and other geometrical and material parameters on basic mechanical characteristics. Despite the high scatter of the results, mainly due to uncertainty as to the real thickness, strain rate, and definition of failure, it was found[5,7,9,10] that the shear strength and modulus are highly variable below a thickness of about 0.2 mm but become invariant at larger thicknesses. In the same context, a resolution to this complexity was sought[9,11] through the mechanical testing of the bulk adhesive, felt to be permissible in view of the fair correlation found to exist between mechanical properties of the bulk and the *in-situ* adhesive. In parallel, advantage was taken of the fact that characterization of the material under uniaxial loading and stress is simple both in practice and in analysis. Moreover, representation of the non-linear stress–strain behaviour of the adhesive by a simplified elasto-plastic model (see Figure 3) was justified by a previous numerical study,[12] which showed similar stress–distribution patterns in the critical boundary zones close to the adhesive edge. Hence it was concluded that two basic parameters suffice as input for approximate stress analysis within a bonded adhesive layer, namely: the initial elastic moduli, and the yield stress which determines the onset of plastic behaviour.



## EXPERIMENTAL

1. *Preparation of specimens*

The adhesive chosen as representative of the ductile structural film type was FM 73 (American Cyanamid Co.). The adherend was aluminum 2024/T3, and the primer—BR-127.

The specimens were of four types (Figure 1):

U1—for uniaxial tensile testing of the bulk adhesive.
U2—for uniaxial compressive testing of the bulk adhesive.
S1— for shear testing of the bulk adhesive.
S2— for shear testing of the *in-situ* adhesive.

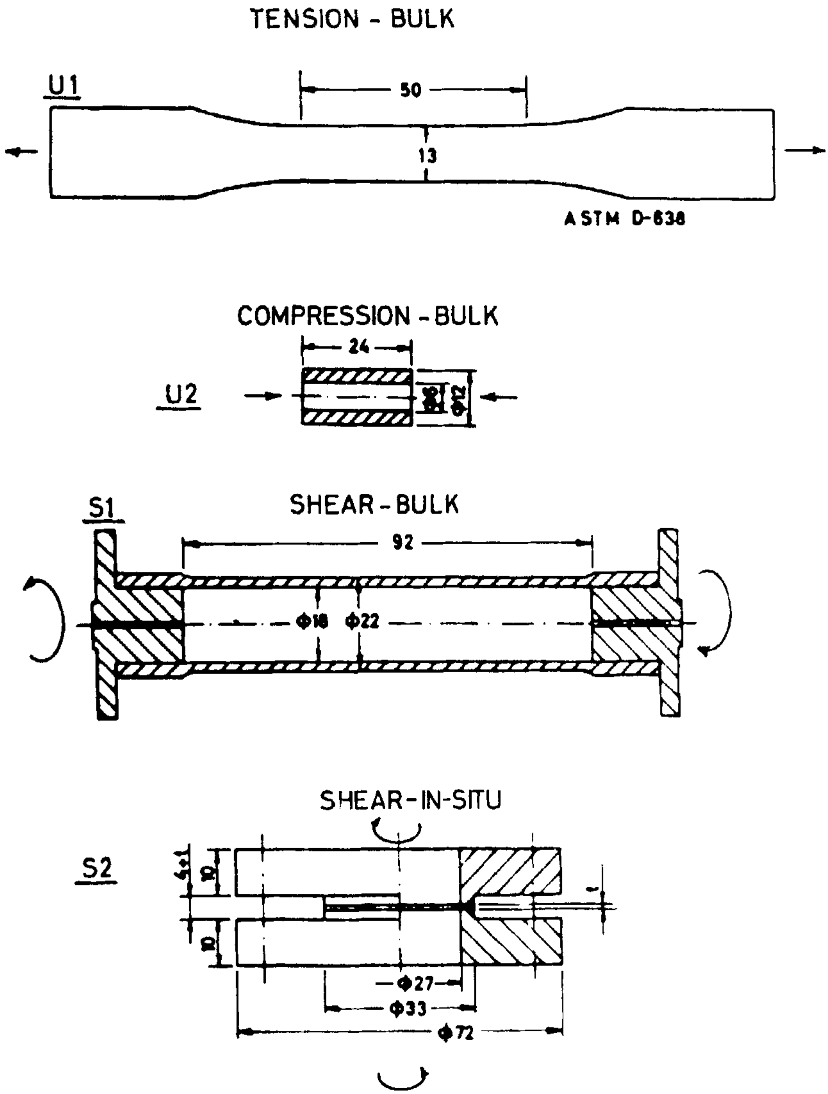


FIGURE 1 Specimen geometry.

The production processes were based on:

(A) *Process A: Unconfined bonding,* ("A"), which permits the free flow of the adhesive along the surface and simulates the state of an adhesive close to the free edges during the bonding process of real structural parts. Production followed the manufacturer's instructions, with film rings compressed between ring adherends. In such a process exact timing is vital but difficult to control; this was demonstrated later by the larger scatter in the final thickness and the mechanical properties of the cured adhesive.

(B) *Process B: Confined bonding*, ("B"). This simulates the state of the adhesive away from the free edges, where hydrostatic pressure prevails prior to the curing stage.

Production comprised the following stages:

—Lay-up of the adhesive films in a closed mould composed of two thick aluminum plates, to prevent flow under pressure.
—Introduction of the closed mould into a press at 10 kg/cm$^2$ pressure.
—Heating to 120°C in 30 minutes.
—Storage at 120°C and 10 kg/cm$^2$ for one hour.
—Slow cooling to room temperature.
—Release of the pressure.
—Machining of the bonded product to the required specimen shape.

The resulting product was superior to its "A" counterpart in both quality and uniformity.



2. *Test procedure*

The S2 specimens were tested on a specially-designed torsional device (Figure 2). The shear strain, $\gamma$, was determined from the corresponding circumferential displacement, the contribution of adherend displacement being deducted with the aid of a control specimen having a "zero thickness" adhesive layer.

The bulk adhesive was tested under torsion on tube specimens (S1) (see Ref. 19), and under tension and under compression (U1, U2, respectively) by standard methods used for polymeric materials, maintaining a constant strain rate ($\dot{\varepsilon}$), the same in all cases. The shear strain rate ($\dot{\gamma}$) was also kept as constant as possible, and related to $\dot{\varepsilon}$ by the ratio: $\dot{\gamma} \simeq \sqrt{3}\,\dot{\varepsilon}$. This is based on the effective stress–strain relationship according to Von Mises' postulate as discussed in Ref. 19.

## RESULTS AND DISCUSSION

Representative stress–strain patterns in tension, compression, and shear, are shown in Figure 3. In most cases a typical ductile mode of behaviour was found, with three distinct ranges:

1) elastic range,
2) non-linear visco-elastic range,
3) visco-plastic yield plateau, characterized by an almost constant stress level, which tends to drop at higher strains.

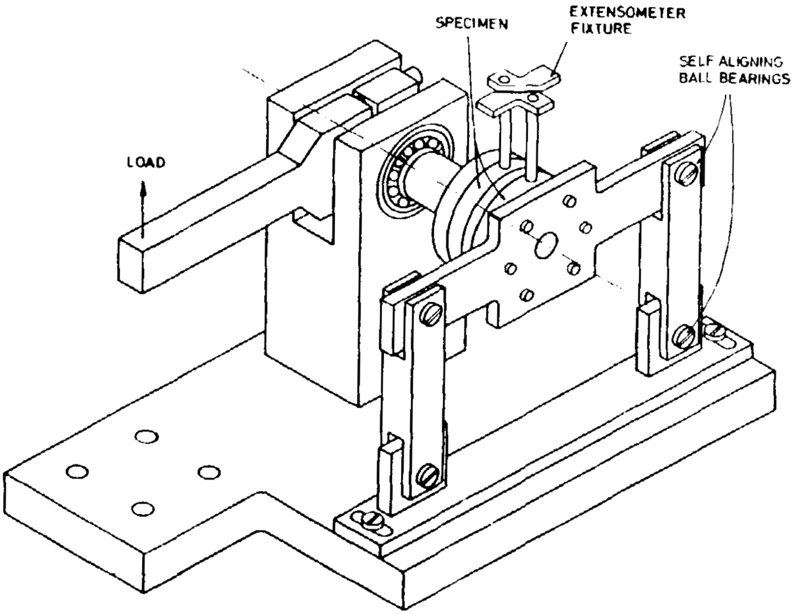


FIGURE 2 Device for the torsional loading of ring specimens.



The yield plateau commenced in the strain range of $\varepsilon_y = 4–5\%$ in tension and compression, and at $\gamma_y = 7–8\%$ in shear. The ratio $\gamma_y/\varepsilon_y = \sqrt{3}$ is in agreement with the ratio expected according to the distortional energy failure criterion of Von Mises' hypothesis.

The discrepancy between bulk and *in-situ* stress–strain curves in shear shown in Figure 3 may be attributed to the variability of *in-situ* characteristics of adhesives as previously discussed.

In the present work the stress at yield initiation is defined as the yield strength of the adhesive under the different loading modes, as follows:

$$\sigma_{yt} = F_t \rightarrow \text{tensile yield strength}$$

$$\sigma_{yc} = F_c \rightarrow \text{compressive yield strength}$$

$$\tau_y = F_s \rightarrow \text{shear yield strength.}$$

## Effect of *in-situ* adhesive thickness on its mechanical characteristics

The average shear strength and shear modulus derived from the stress–strain curves of Type S2 "A" specimens are given in Table I.

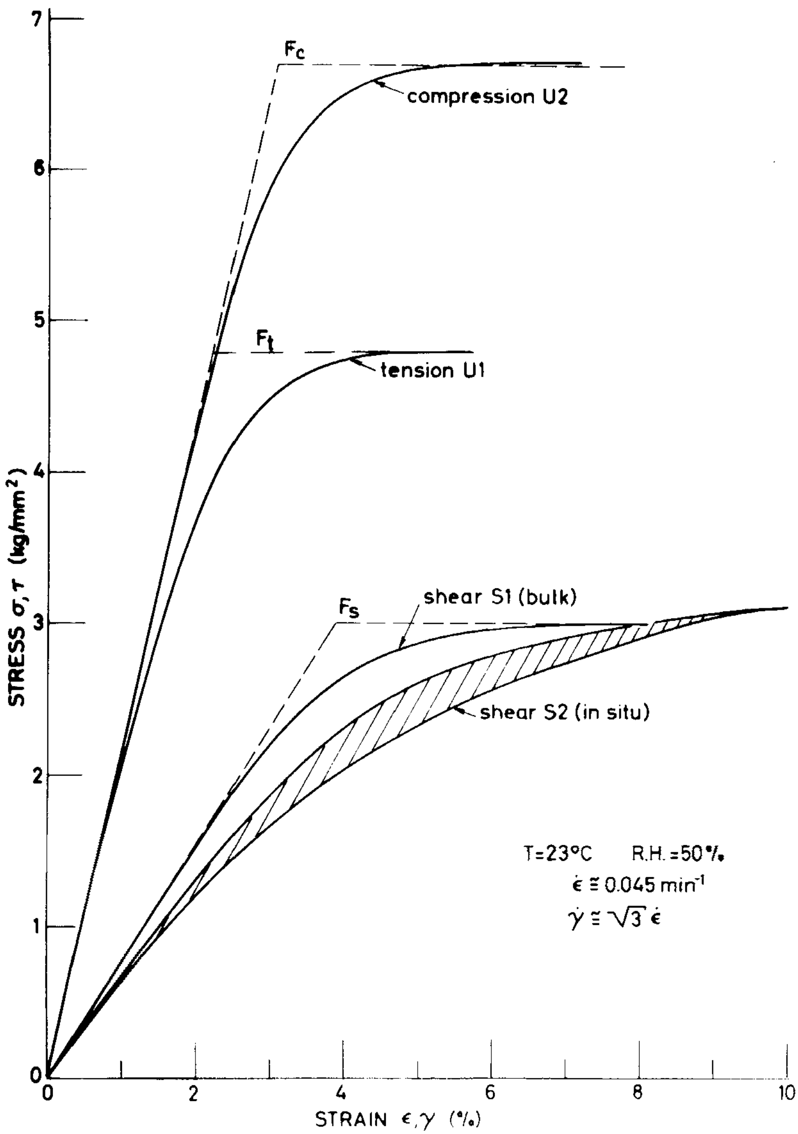


FIGURE 3 Typical stress–strain relationships in compression, tension, and shear, with simplified elasto-plastic representation, of FM 73 adhesive.

TABLE I

Average data of modulus and yield strength from shear tests on *in-situ* "A" specimens

| Thickness range (mm) | Number of specimens — | Average shear modulus $G$[kg/mm$^2$] | Coefficient of variation [%] | Av. shear strength $F_s$[kg/mm$^2$] | Coefficient of variation [%] |
|---|---|---|---|---|---|
| 0.10–0.24 | 8 | 65.2 | 17.6 | 3.00 | 4.30 |
| 0.25–0.49 | 6 | 57.6 | 11.6 | 2.58 | 7.80 |
| 0.5–2.00 | 8 | 70.0 | 7.5 | 2.86 | 9.40 |
| All Popul. 0.1–2.0 | 22 | 65.0 | 14.6 | 2.85 | 9.50 |

In spite of the high scatter the general trend is obvious. The average values for the thinner layers are almost the same as for the whole population; it may thus be concluded that the averages evaluated for the practical thickness range (0.1–0.2 mm) are representative of the whole and that thickness has no significant effect on the elastic and strength characteristics of the adhesive layer. This conclusion, together with other findings, which will be reported in the near future, provide additional support for the assumption that the adhesive layer can be treated as another lamina within a composite laminate.

Corresponding data derived from tests on the tube and the ring "B" specimens were characterized by higher mechanical properties and lower scatter (Table II).

The shear modulus $G$ was obtained from thickness measurements of the *in-situ* layer. The thickness ranged from 0.15 to 0.20 mm, while the measurements entail an inaccuracy of about 5% (not counting the contribution of the primer).

## Experimental *vs.* theoretical prediction

The average moduli and yield strengths of the "B" specimens for two temperatures are given in Tables III and IV respectively.

The predicted shear modulus is

$$G = \frac{E}{2(1+\nu)}. \tag{1}$$

This is based on bulk data and the elastic relationship for isotropic materials. It is in fairly good agreement with its experimental counterpart.

It is also apparent that a relatively high $\lambda$ ratio exists between the compressive and the tensile strength values, a trend similar to that found by other authors[13–16] for polymeric materials. Since this finding conflicts with the basic assumption of the Von Mises failure criterion, according to which the yield strength of a ductile material is unaffected by the isotropic component of the stress tensor, modified criteria have been proposed[17,18] incorporating the

TABLE II

Average shear properties from test on "B" specimens

| Specimen | Average shear modulus $G$ [kg/mm$^2$] | Average shear strength $F_s$ [kg/mm$^2$] |
|---|---|---|
| Tube, bulk S1 | 80 | 3.00 |
| Ring, *in-situ* S2 | 75 | 3.15 |

TABLE III

Experimental and predicted moduli of "B" specimens

| Temperature | Young's modulus in tension $E$ [kg/mm$^2$] | Poisson's ratio $\nu$ | Shear modulus, Experimental $G$ [kg/mm$^2$] Bulk | Shear modulus, Experimental $G$ [kg/mm$^2$] *In-situ* | Shear modulus, Computed (Eq. 1) $G$ [kg/mm$^2$] |
|---|---|---|---|---|---|
| 23°C (RT) | 225 | 0.43 | 80 | 75 | 78.7 |
| 60°C | 145 | 0.40 | 60 | 55 | 51.8 |

TABLE IV

Experimental and predicted yield strengths of "B" specimens

| Temperature | Compressive yield strength $F_c$ [kg/mm$^2$] | Tensile yield strength $F_t$ [kg/mm$^2$] | $\lambda = \frac{F_c}{F_t}$ | Shear yield strength, Experimental $F_s$ [kg/mm$^2$] Bulk | Shear yield strength, Experimental $F_s$ [kg/mm$^2$] *In-situ* | Shear yield strength, Computed (Eq. 7) $F_s$ [kg/mm$^2$] |
|---|---|---|---|---|---|---|
| 23°C (*RT*) | 6.6 | 4.7 | 1.40 | 3.00 | 3.15 | 3.16 |
| 60°C | 3.5 | 2.8 | 1.25 | 1.65 | 1.70 | 1.80 |

isotropic component ($\sigma_m$), related to the octahedral stress ($\tau_{oct}$) by two material constants. For the present case, a linear combination of these variables is proposed, as follows:

$$k_s \tau_{oct} + k_v \sigma_m = 1 \tag{2}$$

$$\tau_{oct} = \frac{1}{3}[(\sigma_1-\sigma_2)^2+(\sigma_2-\sigma_3)^2+(\sigma_3-\sigma_1)^2]^{1/2} \tag{3}$$

$$\sigma_m = \frac{1}{3}[\sigma_1+\sigma_2+\sigma_3] \tag{4}$$

where

$\sigma_1, \sigma_2, \sigma_3$ principal stresses,

$k_s, k_v$ the material constants responsible for the yield due to the distortional and isotropic stress components, respectively.

Equations (2), (3) and (4), are discussed more extensively, and examined experimentally, in a previous paper.[19]

After solving for the particular cases of pure tension, compression, and shear, the following expressions are obtained for the material constants:

$$k_s = \frac{3(\lambda+1)}{2\sqrt{2}\lambda F_t} \tag{5}$$

$$k_v = \frac{3(\lambda-1)}{2\lambda F_t} \tag{6}$$

Substituting of (5) and (6) in (2) and (3) yields a relationship between the shear and the tensile yield strengths, as follows:

$$F_t = \frac{\sqrt{3}}{2}\frac{(\lambda+1)}{\lambda}F_s = \alpha_s F_s \tag{7}$$

Good correlation was found between the shear yield strength values predicted by Eq. (7) and the direct test values for the bulk and the *in-situ* specimens (Table IV). This is also demonstrated by Figure 4, which describes the theoretical relationship between $\alpha_s$ and $\lambda$ versus the relevant experimental data. The fair correlation shown by Figure 4 appears to confirm the general trend predicted by Eq. (7).

The failure envelope according to Eq. (2) is shown in Figure 5. Its spatial shape is conical, the slope decreasing with decreasing $\lambda$. At elevated temperatures with $\lambda$ approaching unity, a cylindrical envelope is obtained in accordance with the classical Von Mises criterion.

## CONCLUSIONS

1) The stress–strain relationship of adhesives represents a non-linear mode of behaviour and may be simulated by a simple elasto-plastic model.

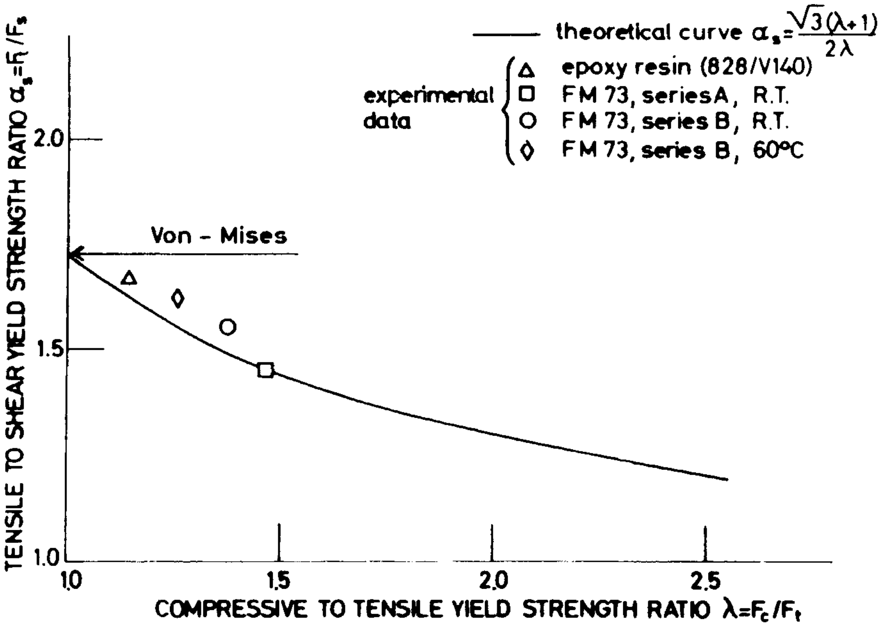


FIGURE 4 $\alpha_s$ and $\lambda$ relationships for several adhesives.

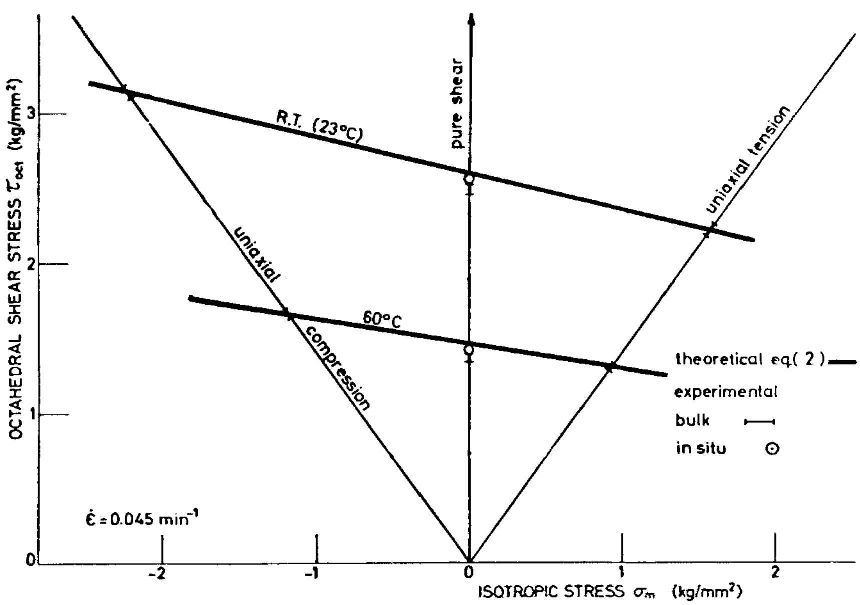


FIGURE 5 Failure envelopes of FM 73 adhesive under different loading and temperature conditions.

2) The mechanical properties of the *in-situ* adhesive and the bulk adhesive material are in fair correlation in the elastic and yield-plateau ranges.

3) The thickness of the adhesive layer seems to have a small effect on its yield strength and elastic moduli (for thicknesses of 0.1 mm and above).

4) The elastic behaviour and the ductile failure modes under a compound stress may well be related to corresponding behaviour of the adhesive layer under uniaxial stress. Such a relationship may be covered by a ductile failure hypothesis incorporating the isotropic stress component.

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