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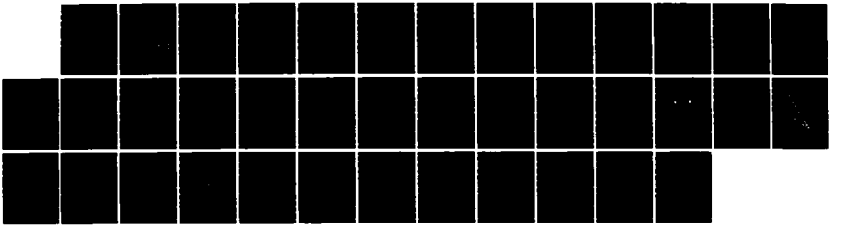

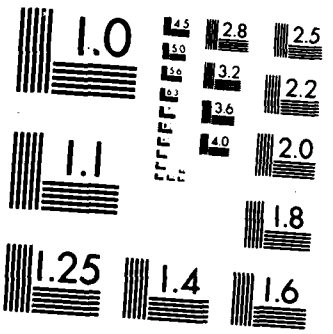

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BLOW-OFF PRESSURES FOR ADHERING LAYERS

by

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An analysis is given of the critical internal pressure P at which a circular debond ("blister") will grow in size, in terms of the tensile modulus E and thickness t of an adhering layer, and the strength G_a of its adhesion to a rigid substrate. Measurements of blow-off pressure are reported for adhering layers of pressure-sensitive tapes having widely-different effective modulus and thickness, and with blisters having a range of diameters. Satisfactory agreement is obtained with the theoretical

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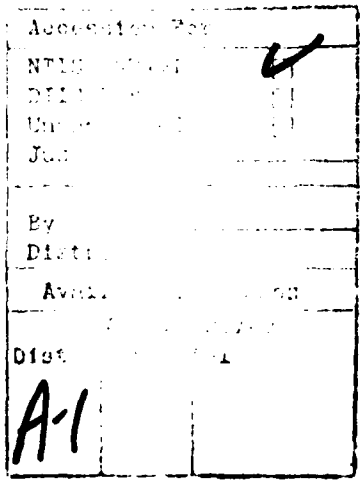
predictions, suggesting that the theory is basically correct in assuming that relatively thin layers behave like elastic membranes. Attention is drawn to the unusual form of the dependence of the debonding pressure P upon the resistance Et of the layer to stretching and upon the detachment energy G_a: $P^4 \propto Et\ G_a^3$. Even though the adhering layer is assumed to be linearly-elastic, the markedly non-linear (cubic) relation between pressure P and volume V of the blister, or maximum height y, leads to this unusual result. The detachment energy is given by a particularly simple function of the pressure P and maximum deflection y of the blister: $G_a = 0.65\ Py$, independent of the stiffness of the adhering layer and diameter of the blister.



1. Introduction

A pressurized blister test is a possible way of measuring the strength of adhesion between a deformable adhering layer and a rigid substrate. It was recommended by Dannenberg (1) and adopted by Williams and colleagues (2, 3) and Andrews and Stevenson (4) to study adhesion in selected systems. Interpretation of the measurements is not a simple matter, however. Three experimental situations can be distinguished: (i) the blister diameter is much smaller than the thickness of the adhering layer, (ii) the blister diameter is comparable to the thickness of the adhering layer, and, (iii) the blister diameter is much larger than the thickness of the adhering layer. Correspondingly, there are three different principal modes of deformation in the pressurized layer: (i) mainly in highly-stressed regions around the edge of the blister diameter, (ii) mainly in bending deformation of the adhesive layer, regarded as a flexible circular plate with a built-in edge constraint, and, (iii) mainly in tensile deformation of the adhesive layer, regarded as an elastic membrane.

In each case, by analysing the changes in stored elastic energy that take place as the blister grows and equating them to the energy required to separate the adhering layer from the substrate, values can be obtained for the critical pressure P for growth of the blister. In the first case, the result is (2, 5)

$$\text{(i)} \quad P^2 = 2\pi E G_a / 3a \tag{1}$$

and in the second case (6),

$$\text{(ii)} \quad P^2 = 128\, E G_a t^3 / 9a^4, \tag{2}$$

where E denotes the tensile (Young's) modulus of the adhering layer, G_a is the energy required for detachment per unit of interfacial area (a measure of

the strength of adhesion), $\underline{a}$ is the radius of the blister, and $\underline{t}$ is the thickness of the deformable layer. For the third case, when the blister radius is relatively large compared to the layer thickness, the result, given in the Appendix, is

$$P^4 = 17.4\ EG_a{}^3 t/a^4. \qquad (3)$$

It is surprisingly different in form to the preceding results. The critical pressure is less strongly dependent upon the tensile modulus and thickness of the adhering layer and more strongly dependent upon the strength of adhesion than before. These marked differences arise from the different elastic response of a membrane to internal pressure in comparison with a plate. Deflections of a plate are directly proportional to the applied pressure, whereas deflections of a membrane are proportional to the one-third power of the inflating pressure (7) (it being assumed in both cases that the deflections are small).

In view of the serious consequences of delamination due to pressure in coatings and sealants, it is important to examine the validity of equation 3 thoroughly. Also, as suggested by Hinckley (8), a pressurized blister test may prove to be a good method of measuring interfacial adhesion. A detailed experimental study has therefore been carried out of the elastic deformation and critical debonding pressures for elastic layers adhering to rigid substrates. The layers consisted of commercial adhesive tapes, chosen for their widely-different elastic modulus. They were applied in multiple layers, so that the tensile stiffness of the composite layer could be changed substantially without any change in the strength of adhesion. They were also applied to two different substrates; Plexiglas and Teflon; so that the strength of adhesion could be changed (at least, in principle) without any

change in the elastic properties of the tape. The experimental procedures and results are described below.

2. Experimental

Two commercial pressure-sensitive tapes were employed: A, an electrical tape with an acrylic adhesive layer and a soft vinyl backing, having a thickness of about 0.18 mm (Tape No. 35, 3M Company); B, a packing tape with a biaxially-oriented polypropylene backing, having a thickness of about 0.09 mm (Tape No. 375, 3M Company). They were chosen because they had similar strengths of adhesion to Plexiglas and Teflon but quite different tensile properties. As shown in Figure 1, tape A gave an approximately linear relation between tensile stress and extension over the range 0 to 20 per cent extension whereas tape B underwent plastic yielding at a tensile strain of about 2-3 per cent. Below this strain, however, the stress-strain relation was substantially linear and a value for the tensile stiffness Et per unit width could be estimated. Experimentally-determined values at a rate of extension of 1×10^{-6} s^{-1}, corresponding to the approximate rate of extension in the blow-off experiments described later, were 900 ± 150 N/M for tape A and 105 ± 15 kN/M for tape B. Using the measured thicknesses t, these results correspond to effective values of tensile modulus E of 5.0 MPa and 1.2 GPa, respectively.

The tapes showed some anisotropy in elastic behavior. Tape A was stiffer in the machine direction in comparison with the transverse direction by about 30 per cent, whereas tape B was stiffer in the transverse direction by about 30 per cent. Values for Et given above are averages for the two directions.

A layer of each tape was adhered to a flat plate of Plexiglas containing a central circular depression, about 1 mm deep and having a diameter of 25,

50 or 75 mm. The tape stretched over the circular depression without adhering to its base, so that an initial debond of well-defined shape and size was obtained. The depression was filled with a silicone vacuum grease also, to prevent any adhesion.

For studies of the elastic behavior, a rigid circular clamp was employed to secure the tape against the Plexiglas plate at the edge of the circular depression, Figure 2a. The effective diameter of the elastic membrane was then the same as that of the circular depression. In blow-off experiments this clamping ring was omitted, Figure 2b. Then, at a critical inflation pressure, further debonding took place at the edges of the circular depression. Measurements were made of the diameter, volume and height of the debonded region ("blister") and of the corresponding pressure required to make it grow, as the mean diameter of the blister increased from its initial value to a maximum value of about 75 mm.

The inflation pressure was measured using a mercury manometer for tape A, and a calibrated Bourdon gauge for tape B when the values were considerably higher, approaching 1 atmos. The volume V of the blister was measured by metering the quantity of water injected into the debond through a small hole in the center of the circular depression, Figure 2. The deflection y of the center of the blister away from the undeformed plane was measured with a cathetometer. All measurements were carried out at ambient temperature, about 25°C, and at a rate of inflation of the blister of about 0.3 ml/min corresponding to a rate of growth of the blister radius of the order of 1 mm/min.

Peeling measurements were carried out at a peel angle of 90° and at the same rate, 1 mm/min, in order to determine the detachment energy G_a directly for each tape and substrate combination:

$$G_a = F/w \tag{4}$$

where F is the peel force and w is the width of the tape.

3. Experimental Results and Discussion

Elastic behavior

When the radius of the blister was held constant by a clamping ring, Figure 2a, its volume V was found to be proportional to the deflection y of the center, as shown in Figure 3. Thus,

$$V = C_1 \pi a^2 y \tag{5}$$

where πa^2 is the area debonded and C_1 is an experimentally-determined constant, 0.52, in good agreement with Hencky's theoretical result (7), given in the Appendix, $C_1 = 0.519$.

Experimental relations between inflation pressure P and maximum deflection y are given in Figure 4 for layers of tape A. Several layers were plied together to give a composite membrane with a tensile stiffness that was a simple multiple of the value for a single layer. The layers were secured with a clamping ring, as shown in Figure 2a, to hold the blister radius a constant during inflation. In each case the pressure P was found to be proportional to y^3, as shown in Figure 4, in good agreement with the theory of elastic membranes (see Appendix, equation A.2) and also proportional to the number N of layers plied together.

$$P = C_2' E t y^3 / a^4 \tag{6}$$

From the slopes of the experimental relations, values of the tensile stiffness coefficient Et were calculated by means of equation A.2, using Hencky's value for the coefficient C_2' of 4.75 (7). The results were closely similar for blister radii of 12.5 and 25 mm: Et = 1.01 kN/m; and in good agreement with the value measured directly by tensile experiments on tape A, Et = 0.90 kN/m. Similar measurements with the stiffer tape B gave less satisfactory agreement, however. Values of Et of 45 ± 5 kN/m were deduced from inflation measurements using equation 6, whereas the directly measured value was considerably larger, 105 kN/m. This discrepancy may arise from difficulties in clamping the stiff tape B firmly at the edge of the blister during inflation experiments.

Debonding conditions

Typical experimental relations for tape B between inflating pressure P, maximum deflection y, and radius a, are shown in Figures 5 and 6. Initially, the membrane inflated into a blister with increasing height y with increasing pressure, but with the original radius a_0 of the circular debond. Then, at a critical pressure P_c, further debonding started and the pressure fell continuously as the blister grew in radius.

Actually, a small amount of debonding took place with increasing pressure, so that the radius of the initial blister grew by about 1 mm before the critical pressure was reached. After this, however, further growth of the blister took place with steadily-decreasing pressures, as the theory predicts (see Appendix). The anomalous behavior observed at the start is attributed to weak adhesion at the edges of the original blister, possibly due to entrapment of silicone grease there.

One of the theoretical predictions is that the product Py is a constant, directly related to the characteristic fracture energy G_a for the bond, equation A.9. The broken curve in Figure 5 is of this form, with the constant chosen to give best agreement with the experimental measurements. As can be seen, the experimental results agree reasonably well with the predicted dependence of P on the blister height y. Similarly, the broken curve in Figure 6 is of the theoretical form, equation A.10; Pa = constant; and again the constant has been chosen to give best agreement with the experimental measurements. And again the agreement is relatively good.

On the other hand, less satisfactory agreement was obtained with the softer tape A, as shown in Figures 7 and 8. During debonding the pressure P fell more rapidly as the blister height y and the radius a increased than an inverse proportionality would predict. This is attributed to a dependence of the fracture energy G_a upon the rate of detachment. During the blow-off experiments the effective rate of peeling changed, being initially more rapid and later slowing down, because of the way in which the experiments were conducted. The blister was inflated at a constant rate of volume increase, of about 0.3 ml/min, and not at a constant rate of increase of radius. Peel experiments revealed that the fracture energy for tape A depended strongly upon the rate of peel, increasing by about 50 per cent for a ten-fold increase in rate. Thus, the products Py and Pa would be expected to have larger values initially, and smaller values later, as was observed in the experiments, Figures 7 and 8, because of a continuous decrease in the effective peel rate.

Fracture energies

Average values of the products Py and Pa were obtained from experimental relations like those shown in Figures 5-8. They are listed in Table 1, together with values of the fracture energy G_a calculated from them by means of equations A.9 and A.10, respectively, using experimentally-determined values of the tensile stiffness coefficients Et in the latter case.

In all cases, values deduced for G_a from Pa and Py are seen to be in excellent agreement. They range from about 15 J/m^2 up to about 150 J/m^2, within the general range expected for pressure-sensitive adhesives, and they are distinctly smaller for a Teflon substrate, as would be expected. However, larger values were obtained by peeling strips of the same tapes away from the same substrates at 90°, given in the final column of Table 1. Similar discrepancies were noted before in comparing values of G_a obtained from pull-off experiments at shallow angles with those obtained from 90° peel tests (9). It was suggested then that the severe bending experienced by tapes in peeling at 90° may lead to additional energy being expended in dissipative processes. Further experiments are necessary to decide whether this factor is indeed responsible for the differences in G_a from the two types of detachment.

4. Conclusions

The following conclusions are obtained:

(i) Adhesive layers can be regarded as elastic membranes when a circular debond ("blister") at the interface is pressurized. As a result, the relation between inflation pressure and blister volume or blister height is approximately a cubic one until the blister starts to increase in radius by further debonding.

(ii) When an energy balance is applied to determine the conditions for growth of the blister by further debonding, a particularly simple relation is found to hold between the fracture energy G_a and the corresponding values of debonding pressure P and blister height y:

$$G_a = 0.65\ Py,$$

independent of the radius of the blister or of the stiffness of the adhering layer.

(iii) Qualitatively similar conclusions were reached previously by Hinckley (8). The quantitative differences are discussed in the Appendix.

(iv) Measurements on two pressure-sensitive tapes, adhering to two different substrates, have been compared with the theoretical predictions. Although agreement is generally satisfactory, values deduced for the fracture energy G_a are consistently smaller than those obtained by peeling strips of the same tapes away from the same substrates at an angle of 90°. A similar discrepancy was noted in an

earlier study of detachment at shallow angles (9). It is provisionally attributed to additional energy dissipation in the tape backing when it is bent sharply away from the substrate at 90°.

Acknowledgements

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Appendix

Theoretical relations for the deformation of a circular elastic membrane under a uniform pressure are reviewed below, and then employed to calculate the blow-off pressure for an adhesive layer containing a circular debond.

(i) Elastic deformation

Inflation of a thin circular elastic membrane, clamped at the periphery, has been analyzed by several authors. The results take the form:

$$V = C_1 \pi a^2 y \qquad (A.1)$$

and

$$y = C_2 (Pa^4/Et)^{1/3} \qquad (A.2)$$

where V is the volume of the "blister", y is the deflection of the center away from the membrane plane in the undeformed state, a and t are the radius and thickness of the membrane, E is Young's modulus for the membrane material, P is the inflating pressure and C_1 and C_2 are numerical coefficients whose values depend upon the value of Poisson's ratio ν. Using series expansions, Hencky (7) obtained values of $C_1 = 0.518$ and $C_2 = 0.662$ for $\nu = 0.3$. Using his procedures, values of $C_1 = 0.519$ and $C_2 = 0.595$ are obtained when $\nu = 0.5$, i.e., for incompressible elastic layers, like rubber.

It should be noted, however, that other authors, using different starting points or purely numerical methods, have obtained slightly different values of C_2 than Hencky for $\nu = 0.3$: 0.653, 0.654 (10-13) but the same value when $\nu = 0.5$: $C_2 = 0.595$ (11). When the considerable approximation is made that the inflated membrane takes up the shape of a spherical cap, values of the coefficients are obtained that are at most only about 4 per cent smaller than Hencky's: $C_1 = 0.5$ for $\nu = 0.3$ or 0.5; and $C_2 = 0.640$ or 0.572 for $\nu = 0.3$ or 0.5, respectively (14).

Thus, there is a substantial level of agreement, although not complete, on the elastic deformation of an inflated membrane. In the analysis of debonding mechanics given below the deformation of the membrane is assumed to be that derived by Hencky.

(ii) Blow-off pressure.

An energy criterion for debonding is assumed to hold in which energy ΔW supplied to the system as the circular debond increases in radius by a small amount Δa is equated to energy expended in the debonding process itself. Changes in elastic energy in the membrane must also be taken into account. Thus,

$$\Delta W = \Delta W_1 + \Delta W_2 \qquad (A.3)$$

where the input energy $\Delta W = P\Delta V$, ΔW_1 denotes energy expended in detachment, given in terms of the characteristic energy G_a of detachment per unit area of bond by

$$\Delta W_1 = 2\pi a \, G_a \, \Delta a \, , \qquad (A.4)$$

and ΔW_2 denotes the change in energy stored elastically in the stretched membrane as the radius of the debond increases by an amount Δa.

Input energy ΔW is given by

$$\Delta W \equiv P(\partial V/\partial a)_p \, \Delta a = (10\ PV/3a)\Delta a \qquad (A.5)$$

from equations A.1 and A.2.

On integrating the cubic relation between pressure and volume for a blister of constant radius a, equations A.1 and A.2, the amount of energy stored in the inflated membrane is obtained as

$$W_2 = PV/4. \qquad (A.6)$$

Thus, as the radius of the blister increases by an amount Δa the energy term W_2 changes by an amount:

$$\Delta W_2 = P(\partial V/\partial a)_p \, \Delta a/4 = \Delta W/4. \qquad (A.7)$$

On substituting from equations A.4, A.5 and A.7 in equation A.3, the detachment energy G_a is obtained as

$$G_a = 0.398\ PV/a^2 \qquad (A.8)$$

or

$$G_a = 0.649\ Py. \qquad (A.9)$$

The blow-off pressure is then obtained in terms of the blister radius a by means of equation A.2,

$$P^4 = 17.4\ Et\ G_a{}^3/a^4. \qquad (A.10)$$

The main features of this analysis were recognized by Hinckley in 1983 (8): that the elastic behavior of an inflated blister follows membrane theory; that the relation between pressure P and deflection y will therefore be a cubic one; and

that an energy balance can be applied to determine the conditions for growth of the blister by further debonding. However, the treatment given here differs from that of Hinckley in two respects: the approximation of the shape of the blister by a spherical cap is not made; instead, the detailed analysis of Hencky is employed; and, more importantly, the energy balance given in equation A.3 is used in place of that proposed by Hinckley, which takes the form

$$\Delta W_1 = \Delta W_2 \qquad (A.11)$$

in the present notation, and is thought to be incorrect. As a result, Hinckley obtained the relation

$$G_a = 0.25\ Py \qquad (A.12)$$

in place of equation A.9.

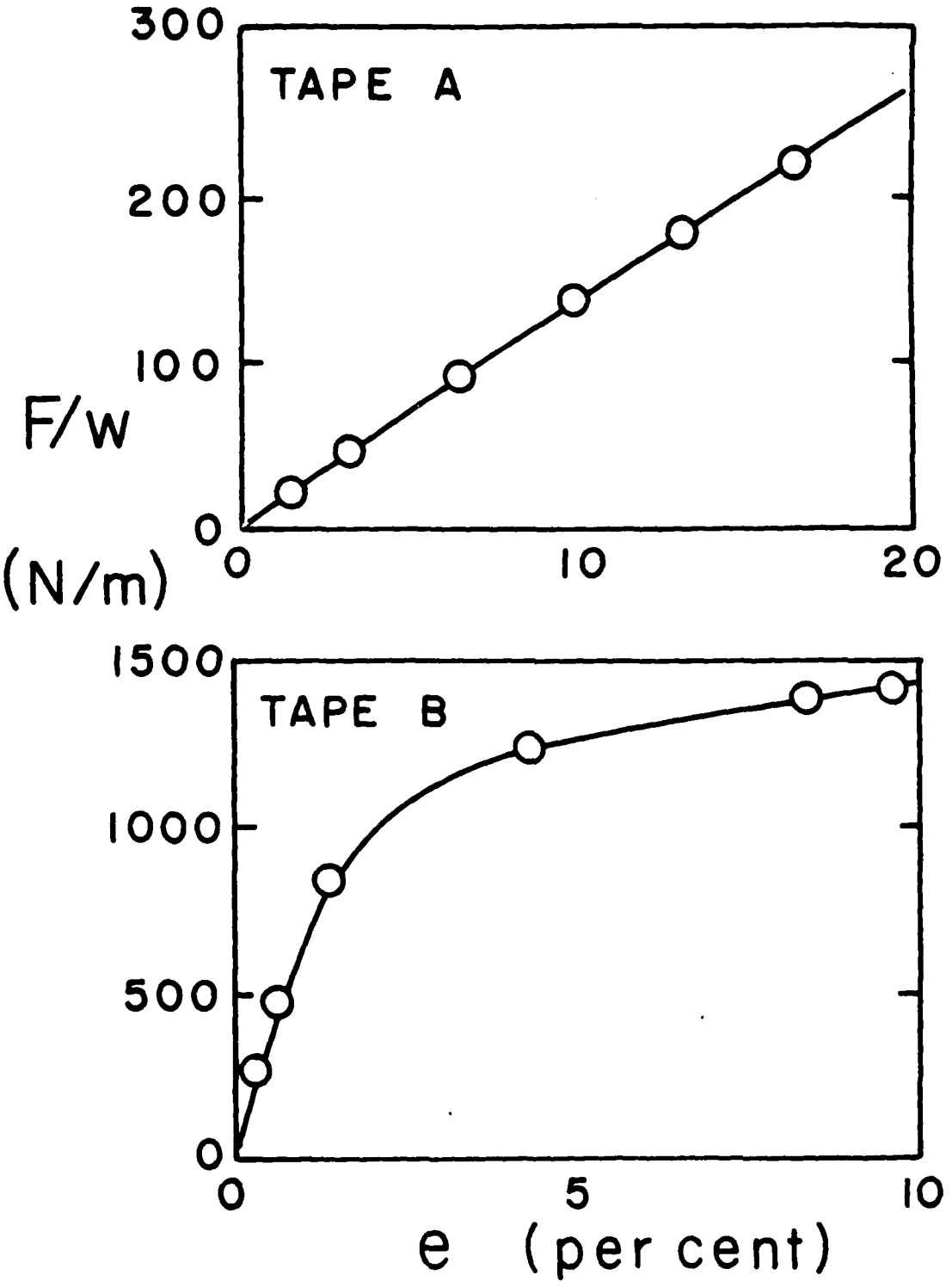
Table 1: Fracture energies G_a (J/m^2) from blow-off and from peeling experiments.

Number N of layers	Pa (N/m)	Py (N/m)	G_a (calc. from Pa)	G_a (calc. from Py)	G_a (from peeling)
			Tape A on Plexiglas substrate		
1	129 ± 11	38 ± 5	26 ± 3	24.5 ± 3	45.2 ± 3
2	155 ± 13	38 ± 3.5	26.5 ± 3	24.5 ± 2.5	
3	165 ± 27	36 ± 7	24.5 ± 5.5	23.5 ± 4.5	
5	175 ± 25	33 ± 6	23 ± 4.5	21.5 ± 3.5	
7	190 ± 15	32 ± 3.5	22.5 ± 2.5	20.8 ± 2.1	
10	195 ± 25	32 ± 5	21 ± 3.5	20.8 ± 3.4	
			Tape A on Teflon substrate		
1	101 ± 7	22.3 ± 2.5	18.5 ± 2	14.5 ± 1.5	46.2 ± 1.5
			Tape B on Plexiglas substrate		
1	1575 ± 65	237 ± 12	150 ± 8	154 ± 8	228 ± 12
			Tape B on Teflon substrate		
1	375 ± 10	41.5 ± 1.5	22.2 ± 1	26.9 ± 1	95.5 ± 6

Figure Captions

1. Relations between tensile force per unit width F/w and extension e for tapes A and B.
2. (a) Measurement of elastic behavior of a pressurized membrane, radius a_o.
   (b) Measurement of blow-off pressures and deflections.
3. Experimental relation between blister volume V and height y for clamped layers having a radius a_o of 38 mm. O, two layers of tape B; ∇, 5 layers of tape A.
4. Experimental relations between inflation pressure P and blister height y for clamped layers of tape A having a radius a_o of 25 mm. N denotes the number of layers plied together.
5. Experimental relation between inflation pressure P and maximum height y of the blister for one layer of tape B. The broken curves are of the theoretical forms: $P \propto y^3$, equation A.2, for inflation; and $P \propto 1/y$, equation A.9, for debonding.
6. Experimental relation between inflation pressure P and blister radius a for one layer of tape B. The broken curve is of the theoretical form, equation A.10, Pa = constant.
7. Experimental relation between inflation pressure P and maximum height y of the blister for one layer of tape A with an initial debond radius $a_o = 12.5$ mm. The broken curve is of the theoretical form; Py = constant, equation A.9.

8. Experimental relation between inflation pressure P and blister radius a for one layer of tape A. The broken curve is of the theoretical form; Pa = constant, equation A.10.


TAPE A
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200
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Figure 1

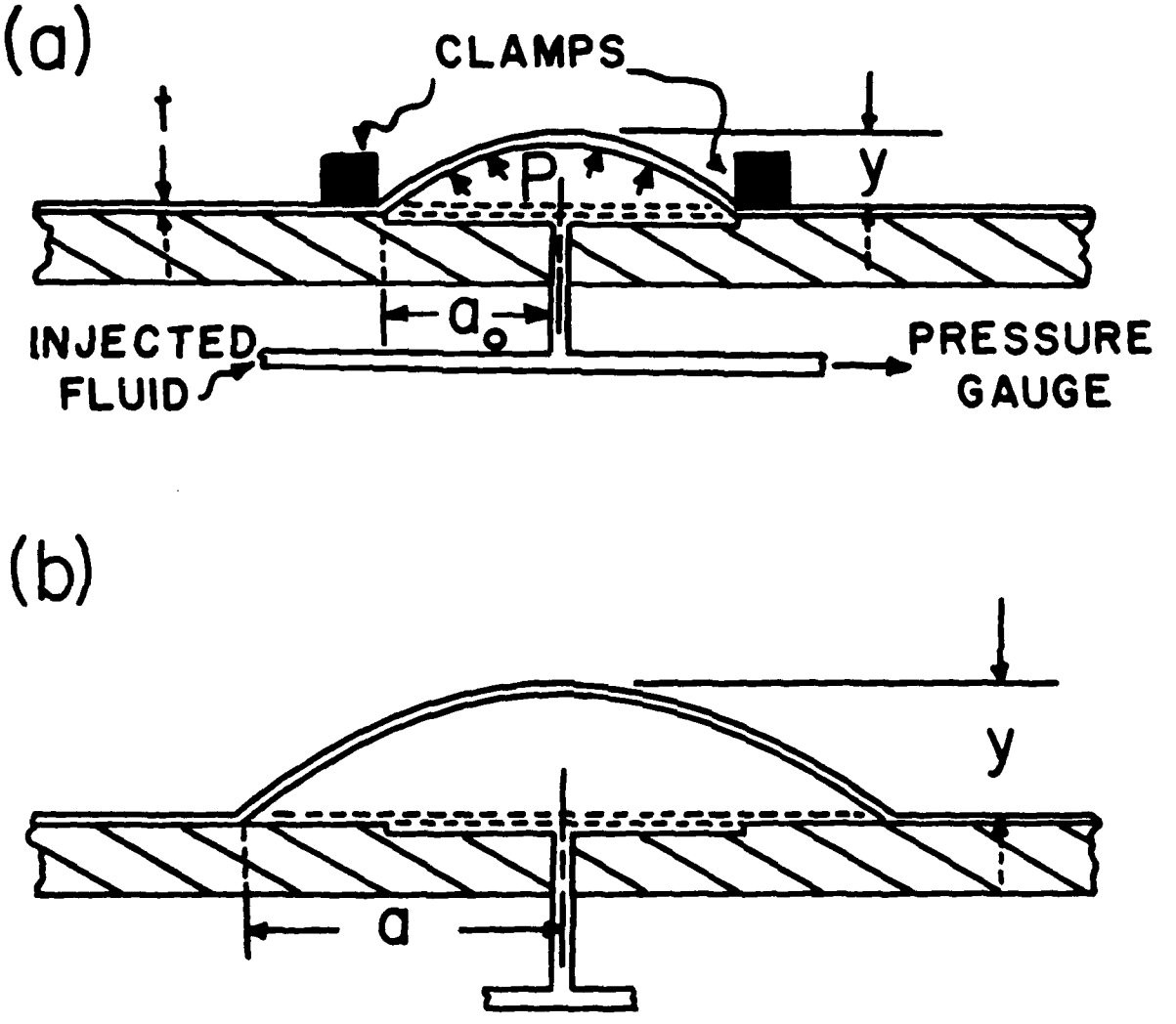


Figure 2

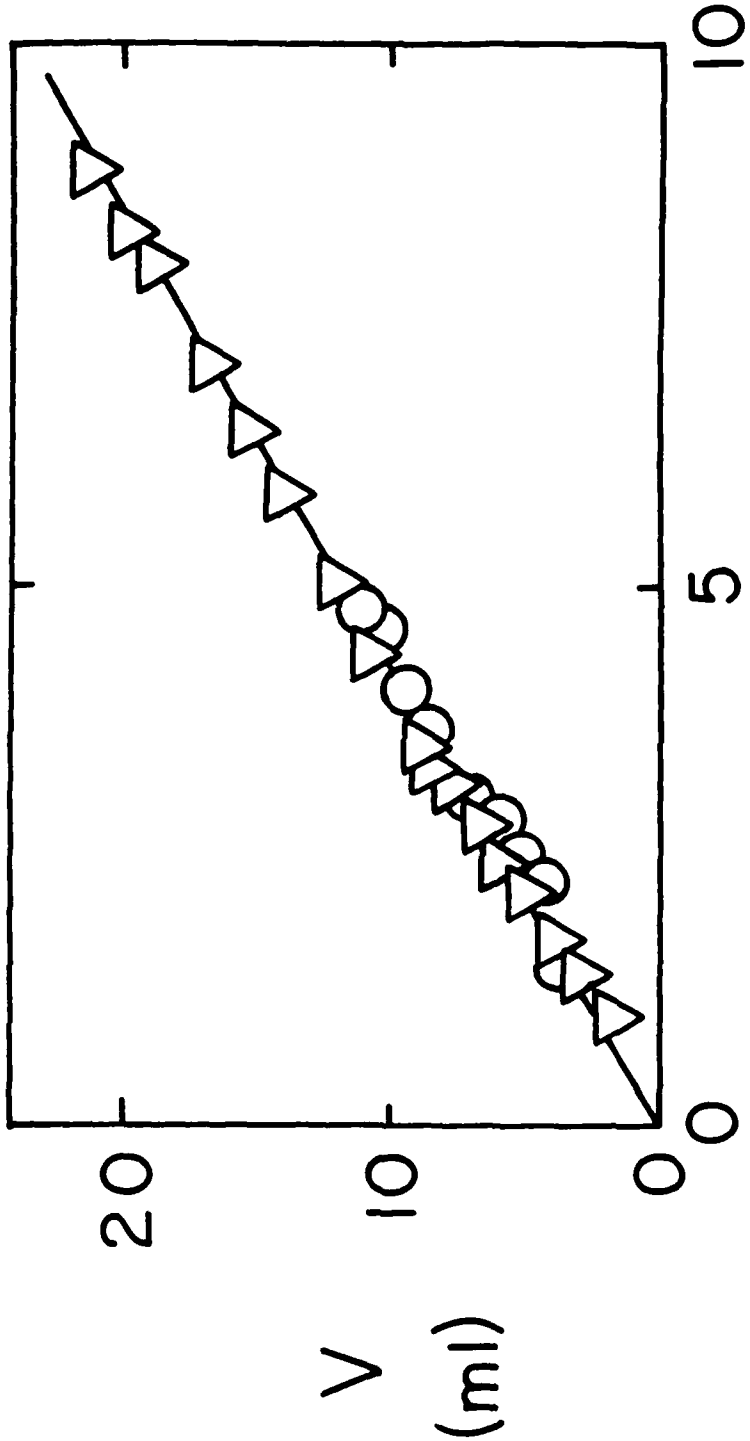
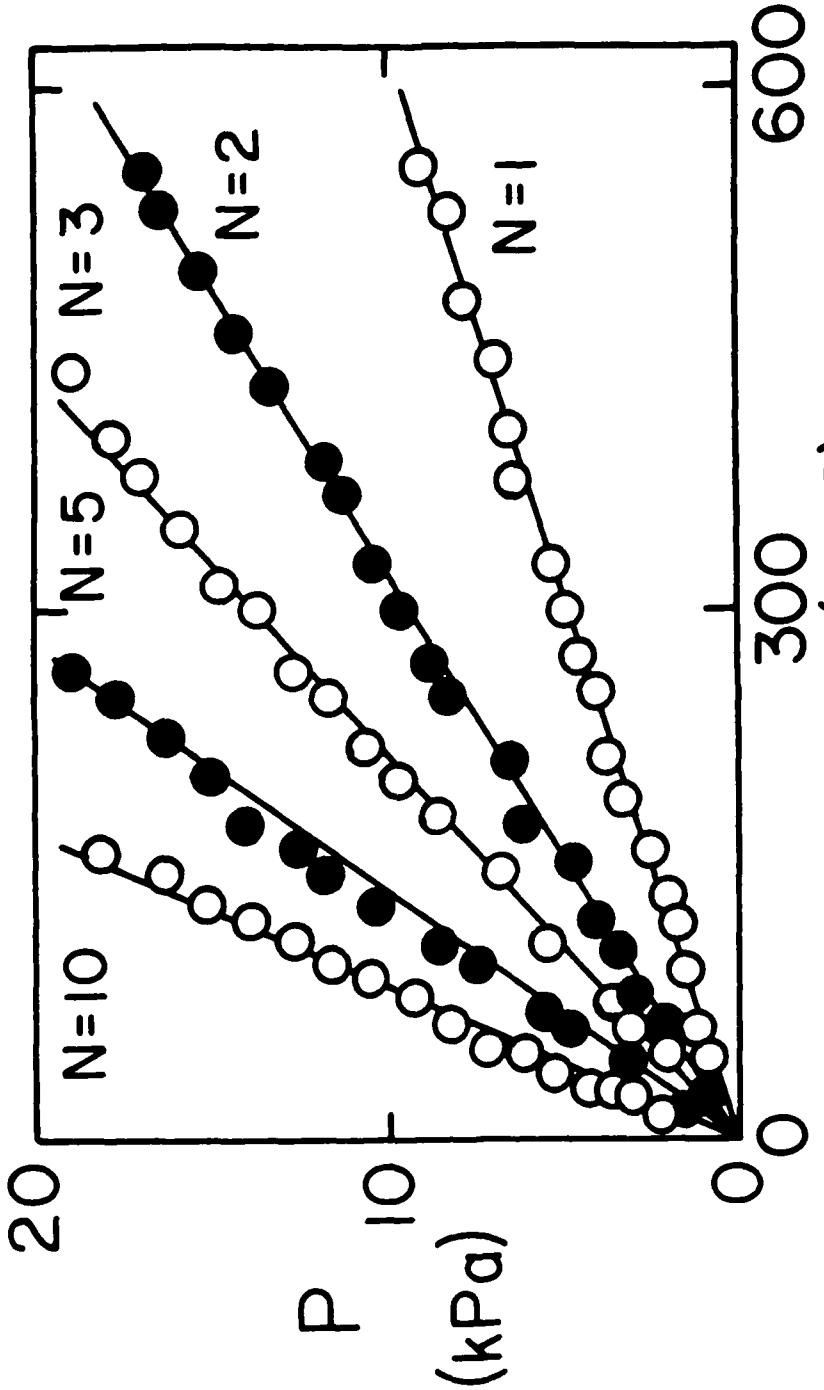


Figure 3



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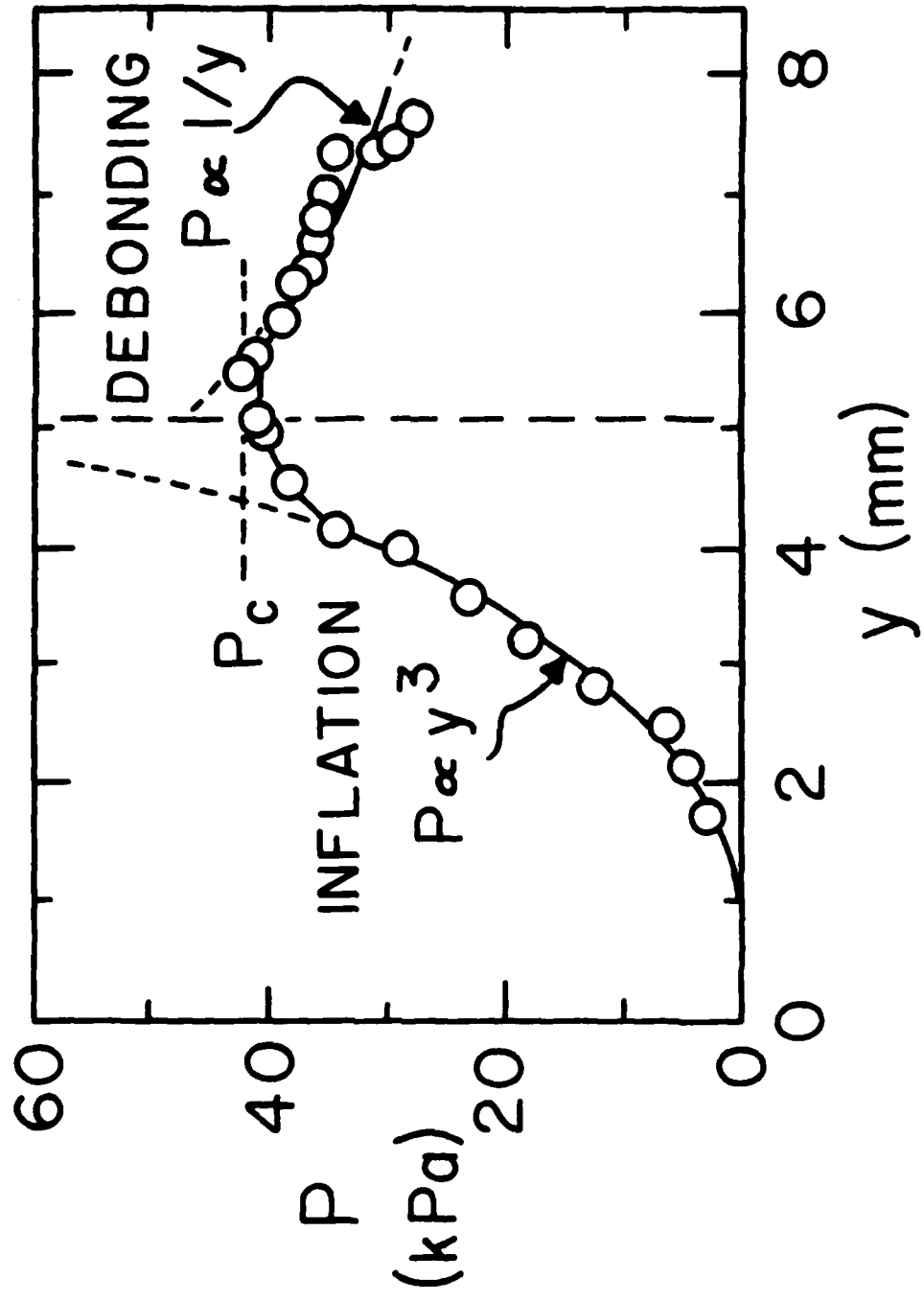

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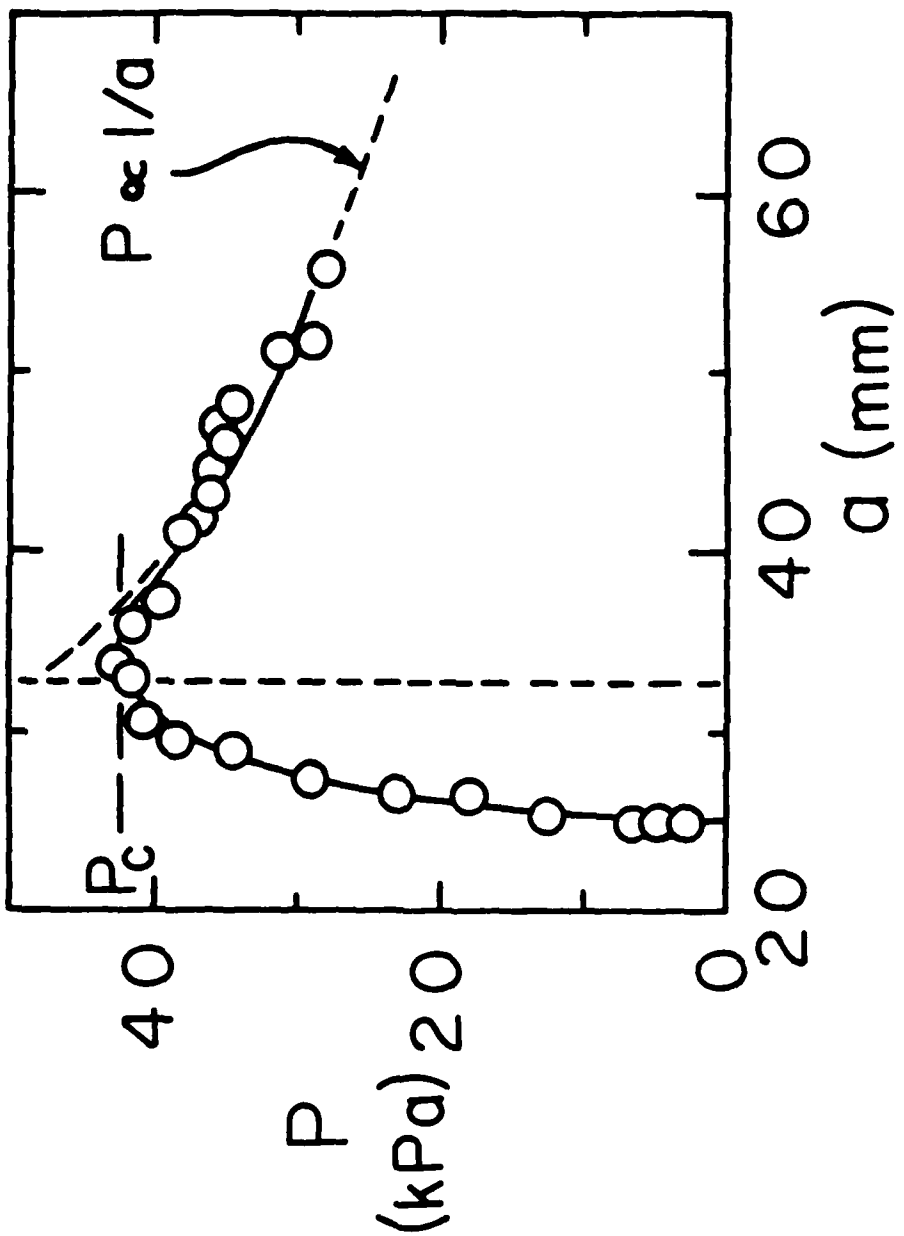


Figure 6

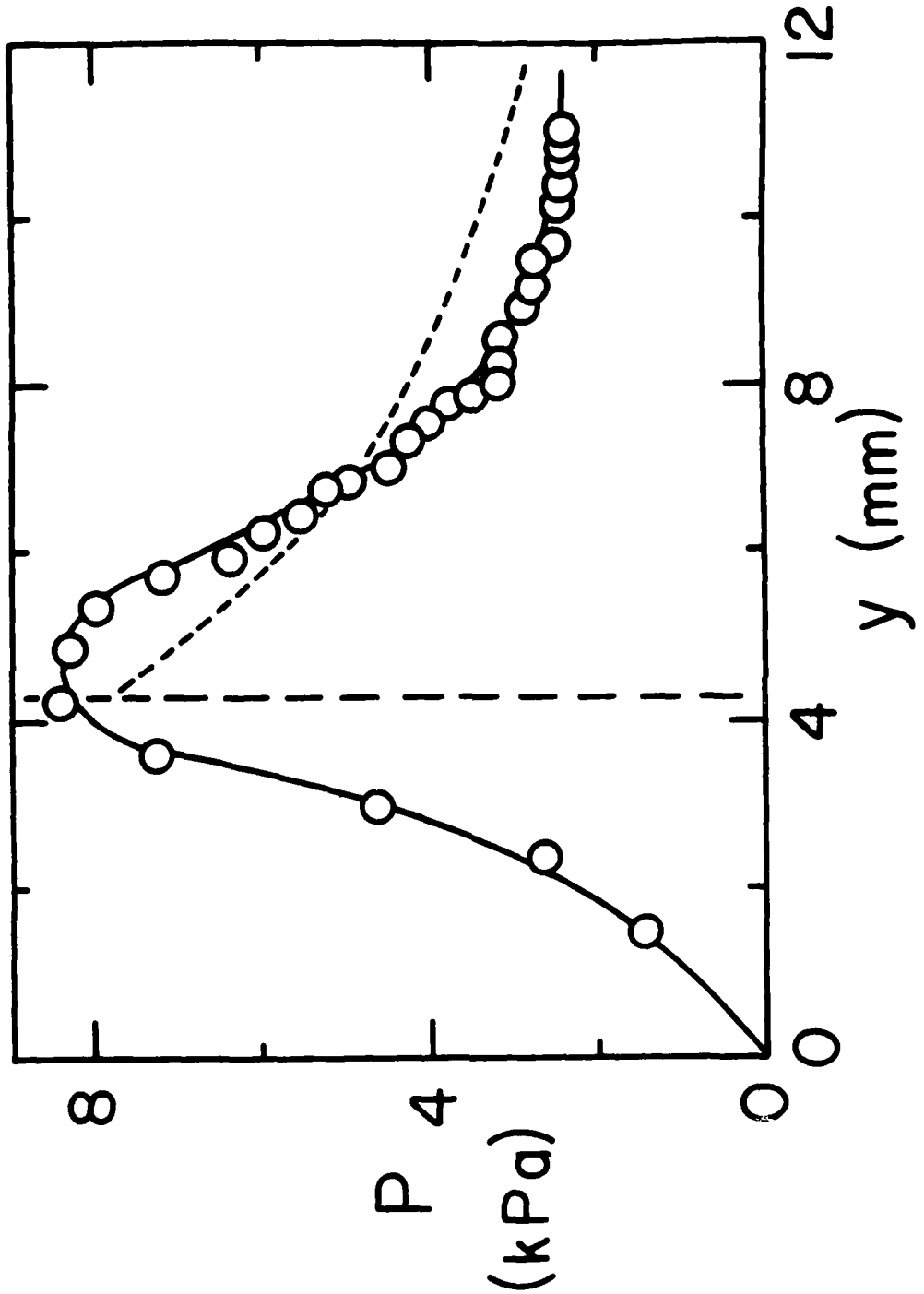
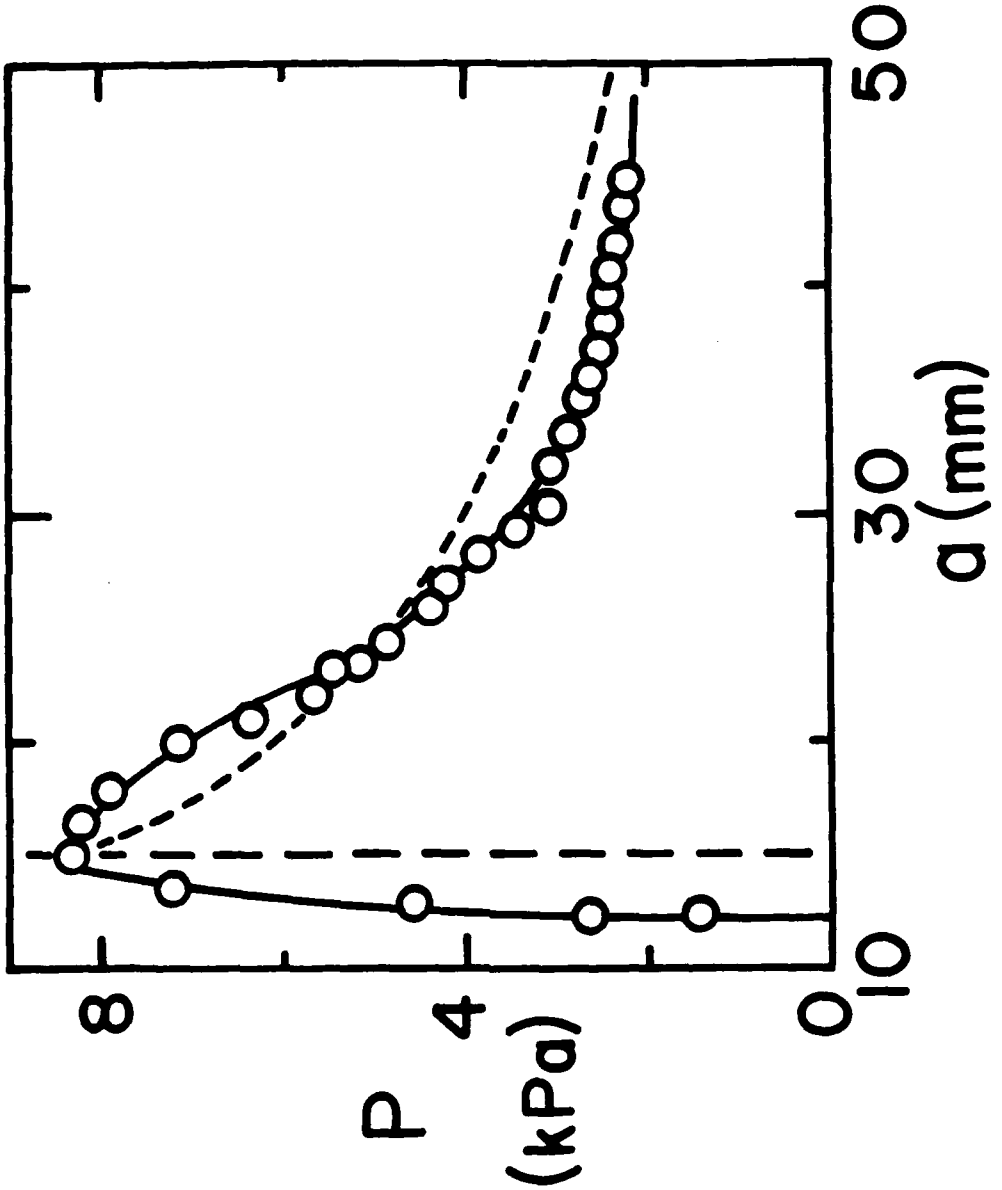


Figure 7



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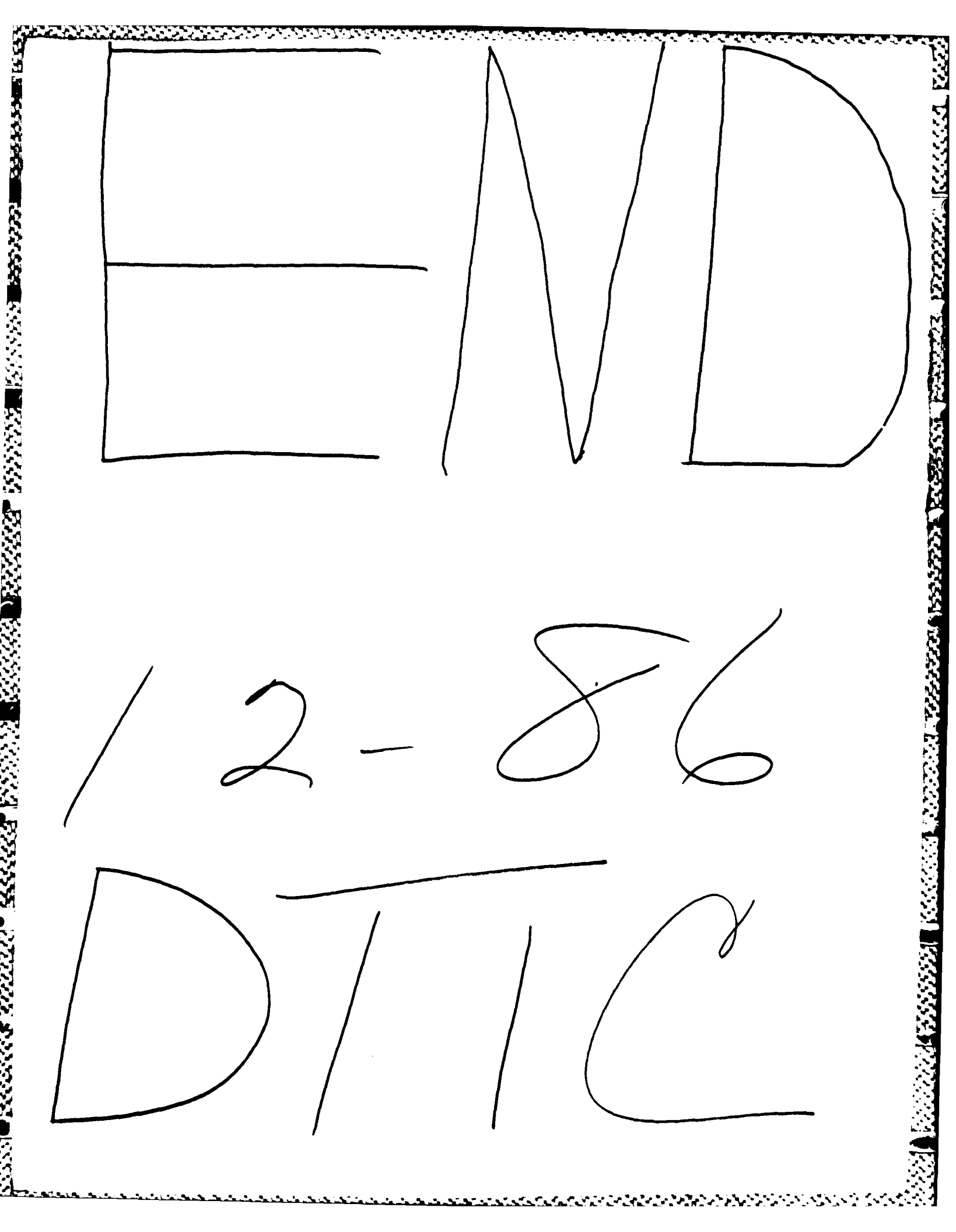
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