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TEAR STRENGTH OF ELASTOMERS UNDER THRESHOLD CONDITIONS

by

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Measurements have been made of the tear strength of several elastomeric materials under threshold conditions, i.e., in the swollen state and at high temperatures when dissipative contributions to the work of fracture are minimized. The materials studied were Neoprene WRT, PB, SBR, EPDM and a castable polysulfide material, LP-32. They were crosslinked to different degrees, using a free-radical source, or sulfur, or a metal oxide as the crosslinking reagent. Values obtained for the threshold tear strength →

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20. (continued)

were similar in all cases, lying in the range 50 - 200 J/m^2. They appeared to be approximately proportional to $E^{-\frac{1}{2}}$, where E is the tensile (Young's) modulus of the elastomer, as predicted by the theoretical treatment of Lake and Thomas. Values for networks with polysulfidic crosslinks were significantly higher than with monosulfide or C-C crosslinks, by about a factor of 2. Values for carbon-black-filled materials were higher also, by about the same factor, in comparison with the equivalent unfilled materials. These effects are attributed to rupture of labile crosslinks, or bonds to carbon black, prior to main-chain rupture.

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Introduction

The tear strength or fracture energy of a material, denoted here G_c, represents the amount of mechanical energy required to propagate a tear through a unit area of the material. Vulcanized rubber compounds have a minimum tear strength under certain conditions, when visco-elastic and other dissipative processes are minimized. This minimum strength, denoted here the threshold strength, has been measured previously for a number of elastomeric networks (1-4). The results have been found to agree satisfactorily with a theoretical treatment due to Lake and Thomas (5) both in absolute magnitude and in the dependence upon the degree of crosslinking (3,4). In accordance with the theory, the threshold tear strength is found to increase with increasing length of the molecular strands comprising the network, approximately in proportion to $M_c^{\frac{1}{2}}$ where M_c is the average molecular weight of a network strand. Thus, the relationship for the threshold tear strength G_{co} is

$$G_{co} = K M_c^{\frac{1}{2}}. \tag{1}$$

The coefficient K relating the threshold tear strength to the molecular weight of network strands is itself dependent upon the chemical structure of the molecules comprising the network. It depends upon the length of the main-chain bonds, the average mass per main-chain atom, the flexibility of the chain and the dissociation energy of the weakest main-chain bond. This dependence has been shown to account for the particularly low threshold strengths of silicone rubber and phosphonitrilic fluoroelastomer compared to polybutadiene and polyisoprene (3,4). The principal factor responsible is

the larger average mass per main-chain atom for the former two elastomers compared to the latter.

Equation 1 can be reformulated in terms of the tensile (Young's) modulus E of elasticity of the material, using the theoretical relation (6)

$$M_c = 3\rho RT/E$$

where R is the gas constant and T is absolute temperature. Equation 1 then becomes

$$G_{co} = K_1 E^{-\frac{1}{2}}, \tag{2}$$

and predicts that the threshold tear strength is a slowly decreasing function of Young's modulus E.

Measurements have now been made of the threshold tear strengths for several other elastomeric materials: a representative copolymer of styrene and butadiene (25:75, SBR 1502, Firestone Tire and Rubber Company; polychloroprene (Neoprene WRT, E. I. Du Pont de Nemours & Co.); a polysulfide sealant material (Thiokol LP-32); and a copolymer of ethylene and propylene (EPDM, Vistalon 4608, Exxon Chemical Company). These materials represent a wide variety of chemical structures.

In addition, measurements have been made of the threshold strength of molecular networks prepared by free-radical crosslinking, when the molecules are interlinked by C-C bonds, and by sulphur crosslinking systems of various kinds, in order to study the effect of crosslink type upon the threshold tear strength.

Finally, some preliminary measurements are reported of the threshold fracture energies for a carbon-black-reinforced elastomer.

Experimental

(a) Preparation of test strips

Mix formulations and vulcanization conditions are given in Table 1. In all cases, vulcanized rubber sheets were prepared, about 1 - 2 mm thick. Test strips were then cut from the sheets, about 2 cm wide and 6 cm long.

(b) Measurement of tear strength

The threshold tear strength is attained when dissipative processes are minimized. Measurements of the tear strength were therefore carried out at high temperatures, $80^{\circ}C$ - $150^{\circ}C$, at low rates of tearing, 1 - 10 μm/s, and using samples swollen with mobile liquids. Test strips were scored along a central line to a depth of about one-half of the thickness, leaving the other half to be torn through, as shown schematically in Figure 1. The tear strength G_c was calculated from the tear force F as follows

$$G_c = 2 \lambda_s^2 \; F/w$$

where λ_s is the linear swelling ratio of the sample and w is the measured width of the tear path (Figure 1b). The factor λ_s^2 takes into account the reduced number of network strands crossing a unit area in the swollen material. For unswollen specimens $\lambda_s = 1$.

Polybutadiene (PB), SBR and EPDM materials were swollen with liquid paraffin oil. Neoprene WRT and Thiokol LP-32 vulcanizates were swollen with 1, 2, 4-trichlorobenzene (TCB).

(c) Measurement of E

Values of E were determined from tensile stress-strain relations at small strains, using unswollen samples.

Results and discussion

Experimentally-determined values of the threshold tear strength G_{co} are given in Table 2 for all of the materials examined. They show several important features, as discussed below.

Polychloroprene materials

It proved possible to crosslink the polychloroprene elastomer with either an oxide crosslinking system or a sulfur crosslinking system, although in neither case was it found possible to obtain a high degree of crosslinking. Values of Young's modulus E were consequently rather low, Table 2. When conventional formulations were used, capable of forming both types of crosslink, substantially higher values of E were obtained.

Difficulties were experienced in all cases in determining the threshold values of tear energy, G_{co}. As shown in Figure 2, when the tear strength G_c of unswollen samples is plotted as a function of temperature, it appears to reach a lower limit at temperatures of about $120^{o}C$ or higher, but these values are unexpectedly high, 150 - 500 J/m^2. Moreover, at temperatures of $150^{o}C$ or higher the samples showed signs of rapid decomposition. Similar behavior was shown previously by cis-polyisoprene materials (4). It is attributed to the occurrence of strain-induced crystallinity at the tip of the propagating tear, even at temperatures as high as $150^{o}C$, which enhances the tear strength markedly.

Values of the tear strength of highly-swollen samples were found to be much lower, comparable to those for other elastomeric materials, and they did not vary significantly with the test temperature over

the range 80°C to 140°C or with the nature of the swelling liquid.
These values have therefore been taken as threshold values, although
no independent evidence of having reached threshold conditions was
obtained.

Polychloroprene, SBR, polysulfide, PB and EPDM

As the results given in Table 2 show, these materials all gave
roughly similar values of threshold tear strength, about 50-100 J/m^2,
when crosslinked with oxide, C-C, or simple sulfur crosslinks. Thus,
there is no obvious effect of the varied chemical structure of the
polymer molecules, or of the crosslinks that connect them, upon the
tear strength.

When the values of threshold tear strength for these materials
were plotted against the corresponding values of Young's modulus E,
Figure 3, using logarithmic scales for both axes, the results were
found to be consistent with a linear relationship having a slope
of -1/2, in accordance with equation 2. Thus, the variations found
in the threshold tear strength for these different materials are
apparently largely accounted for by variations in the degree of cross-
linking. Neither the chemical structure of the polymer chain nor
of the crosslinks between them appears to be significant. This is
probably because of the close similarity in chain flexibility, bond
length, mass per main-chain atom, and bond dissociation energy for
these varied systems. However, other systems were found to be
significantly stronger, as discussed below. It has previously been
shown that polydimethylsiloxanes and phosphonitrilic fluoroelastomers
are substantially weaker, because of their greater mass per main-
chain atom (4).

Sulfur crosslinking

In Table 2, the symbol $-S_n-$ is used to denote crosslinking systems which yield predominately polysulfidic crosslinks, having more than two sulfur atoms per crosslink. (The symbol $-S_x-$ is used to denote systems which yield crosslinks of unknown structure, but probably with two or fewer sulfur atoms per crosslink.)

The "inefficiently" crosslinked materials, with polysulphidic crosslinks, were found to be significantly stronger than those with monosulfidic, disulfidic or C-C crosslinks. At the same general level of crosslinking, and therefore at similar values of Young's modulus, the threshold tear strength was approximately twice as high.

It has been generally accepted that polysulfidic crosslinks give stronger vulcanizates than monosulfidic or C-C crosslinks (7), but the present measurements are the first ones to be carried out under threshold conditions, as far as the authors are aware. Explanations in terms of enhanced energy dissipation are thus ruled out. Instead, it seems probable that crosslink rearrangement or scission before rupture of the main chain is responsible for the observed strengthening feature (7). In effect, the small-strain elastic properties are those of relatively highly-crosslinked, short-chain, vulcanizates while the tear strengths arise from relatively lightly-crosslinked, long-chain structures, as a result of rearrangement of the original crosslinks under high stresses.

Carbon black reinforcement

The determination of the threshold tear strength for carbon-black-reinforced materials was found to be more difficult than for the corresponding unfilled materials. Low and constant values of tear

strength were only attained in the swollen state and at elevated
temperatures. Although these values have been taken to be threshold
values, there was no clear proof that threshold conditions had, indeed,
been achieved.

The values obtained in this way were found to be considerably higher,
about twice as high, as for the corresponding unfilled materials
(Table 2), being generally around 200 J/m^2. Because the reinforced
compounds were based on sulfur crosslinking systems yielding poly-
sulfidic crosslinks (as is customary) the corresponding unfilled
materials were stronger than the simplest vulcanizates, by another
factor of about 2, as discussed previously. Thus, the carbon-black-
reinforced materials were 3-4 times stronger than the simplest
unreinforced vulcanizate of the same elastomer under threshold conditions.

The large reinforcing effect of carbon black under normal conditions
is well-known. It is interesting to note that a substantial degree
of reinforcement remains under threshold conditions, when dissipative
effects are minimized. It must therefore be ascribed to structural
effects. One possible mechanism of reinforcement, analogous to that
put forward in connection with polysulfidic crosslinking, would be
the detachment of adhering polymer molecules from particles of carbon
black at forces somewhat below those causing main-chain fracture.
Thus, again, the effective network strand would be initially short,
giving a stiff material, but at high stresses it would become longer,
and hence give a strong, tear-resistant, material.

Acknowledgements

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Table 1. Mix formulations, in parts by weight, and vulcanization conditions employed for preparing test specimens.

Polychloroprene, oxide crosslinks:

   Neoprene WRT, 100; magnesium oxide, 2; zinc oxide, 5.

   Vulcanized at 150°C for 30, 40 and 90 min.

Polychloroprene, sulfur crosslinks:

   Neoprene WRT, 100; sulfur, 2; diorthotolyl guanidine (DOTG),

   0.5; tetramethylthiuram disulfide, 1.5. Vulcanized at

   140°C for 40 and 80 min.

Polychloroprene, oxide and sulfur crosslinks:

   (i) Neoprene WRT, 100; sulfur, 1; tetramethylthiuram

        monosulfide (TMTM), 1; DOTG, 0.5; magnesium oxide, 4;

        zinc oxide, 5; stearic acid, 1.

   (ii) Neoprene WRT, 100; sulfur 2; TMTM, 1; DOTG, 1;

        magnesium oxide, 8; zinc oxide, 10.

   Both compounds were vulcanized at 150°C for 2 h.

Polysulfide:

   (i) Thiokol LP-32, 100; manganese dioxide, 4.

   (ii) Thiokol LP-32, 100; manganese dioxide, 10.

   Both compounds were vulcanized at 100°C for 1 h.

Table 1 (continued)

SBR:

FR-S 1502, 100; dicumyl peroxide, 0.5. Vulcanized at 150°C
for 2 h.

Polybutadiene (PB), carbon-carbon crosslinks:

Diene 35 NFA, 100; dicumyl peroxide, 0.2. Vulcanized at
150°C for 1 h.

Polybutadiene, EV formulation:

Diene 35 NFA, 100; zinc oxide, 5; sulfur, 0.6; zinc 2-ethyl
hexanoate, 2; 2-morpholinothiobenzthiazole (Santocure MOR),
1.44; tetrabutylthiuram disulfide, 0.6; Agerite Resin D, 1.
Vulcanized at 140°C for 40 min.

Polybutadiene, conventional sulfur recipe:

Diene 35 NFA, 100; zinc oxide, 3.5; stearic acid, 2.5;
sulfur, 2; Santocure MOR, 0.6; Philrich oil HA5, 5;
phenyl-2-naphthylamine (PBNA); 1. Vulcanized at 150°C for
1 h.

Polybutadiene, carbon-black-reinforced, conventional sulfur recipe:

(i) As the preceding unfilled recipe, plus:

N330 carbon black (Cabot Corporation, Vulcan 3), 50.

(ii) As the preceding unfilled recipe, plus:

N765 carbon black (Cabot Corporation, Sterling
black), 50.

Both compounds were vulcanized at 150°C for 1 h.

Table 1 (continued)

Ethylene-propylene copolymer (EPDM), carbon-carbon crosslinks:

Vistalon 4608, 100; dicumyl peroxide, 2. Vulcanized at 160°C

for 2 h.

EPDM, sulfur crosslinks:

Vistalon 4608, 100; sulfur, 1.5; mercaptobenzothiazole, 0.6;

methyl ethyl tuads, 1.5; zinc oxide, 4; stearic acid, 1.

Vulcanized at 150°C for 40 min.

EPDM, carbon-black-reinforced, sulfur crosslinks:

As the preceding unfilled recipe, plus:

N330 carbon black (Cabot Corporation, Vulcan 3), 50.

Vulcanized at 150° for 40 min.

Table 2. Threshold tear strength G_{co} for various compounds. (The mix formulations and vulcanization conditions are given in the Appendix.)

Elastomer	Crosslink type	Young's modulus E (kPa)	Linear swelling ratio λ_s	G_{co} (J/m^2)
			Swollen with TCB	
Neoprene WRT	-O-	370	2.58	120
"	"	610	2.32	110
"	"	850	2.17	66
Neoprene WRT	$-S_x-$	600	2.39	88
"	"	850	2.17	70
Neoprene WRT	-O- and $-S_x-$	2050	1.6	48
"	"	3200	1.4	65
Thiokol LP-32	$-S_2-$	640	1.71	96
"	"	1010	1.67	66
			Swollen with paraffin oil	
SBR 1502	-C-	2250	1.20	60
PB (Diene 35 NFA)	-C-	2900	1.30	62
"	-S-(EV)	-	1.32	56
"	$-S_n-$	-	1.34	106
PB + 50 phr N330 carbon black	$-S_n-$	-	1.2	200
PB + 50 phr N765 carbon black	$-S_n-$	-	1.36	ca 200
EPDM (Vistalon 4608)	-C-		1.38	56
"	$-S_n-$		1.44	ca 160
EPDM + 50 phr N330 carbon black	$-S_n-$		1.3	210

<u>Figure captions</u>

Figure 1.   Method of measuring tear strength.

Figure 2.   Tear strength G_c vs temperature for polychloroprene and
SBR vulcanizates. Polychloroprene oxide crosslinks:$\triangle,\diamond$;
sulfur crosslinks: $+,\oplus,\square$. SBR, C-C crosslinks: O

Figure 3.   Threshold tear strength G_{co} vs Young's modulus E.
Polychloroprene materials, O; polysulfide, $\oplus$; polybutadiene,
$\bullet$; SBR, $\oplus$. The line is drawn with a slope of $-\frac{1}{2}$.

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FIGURE 1

FIGURE 2

Log₁₀E (kPa)
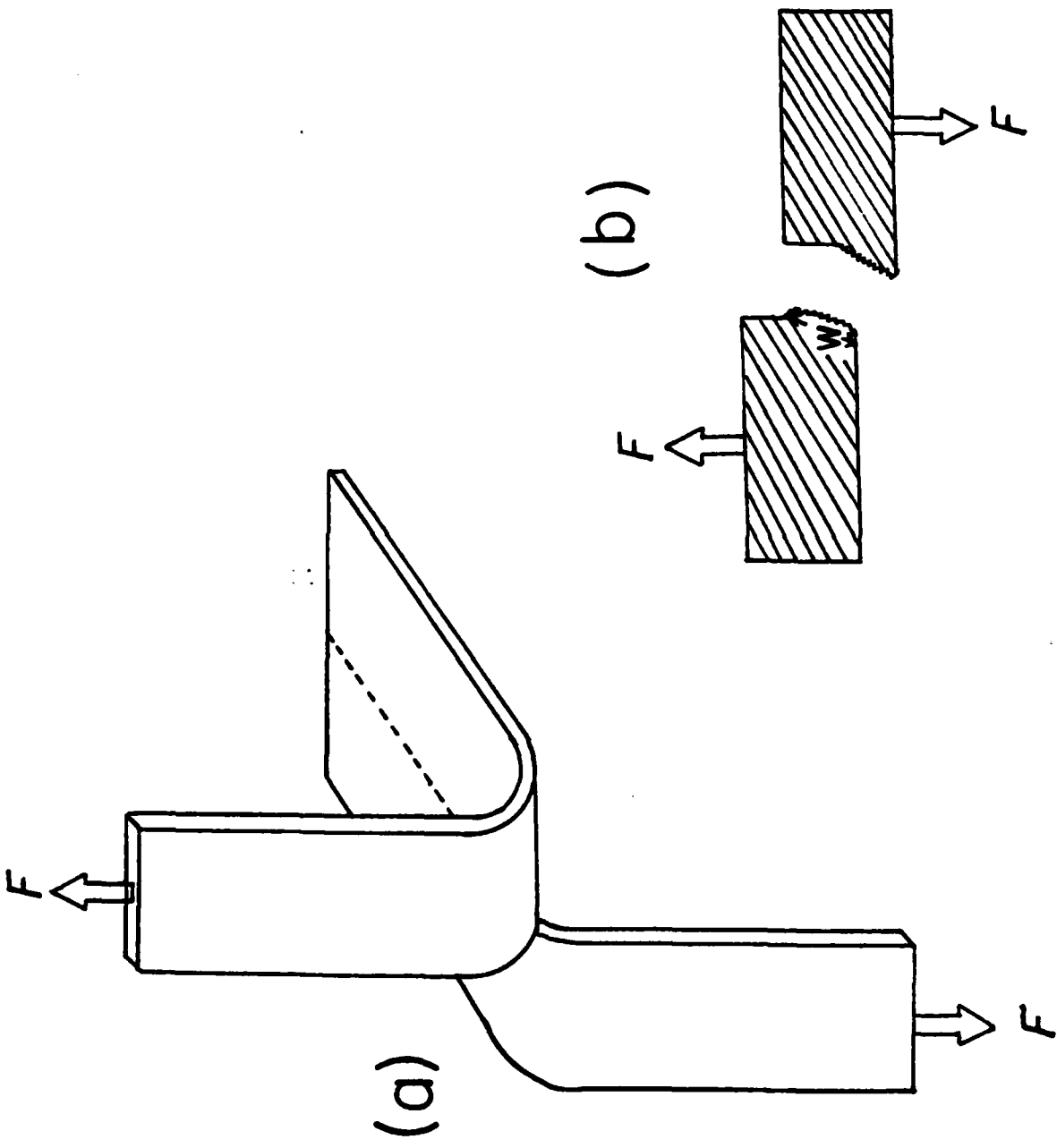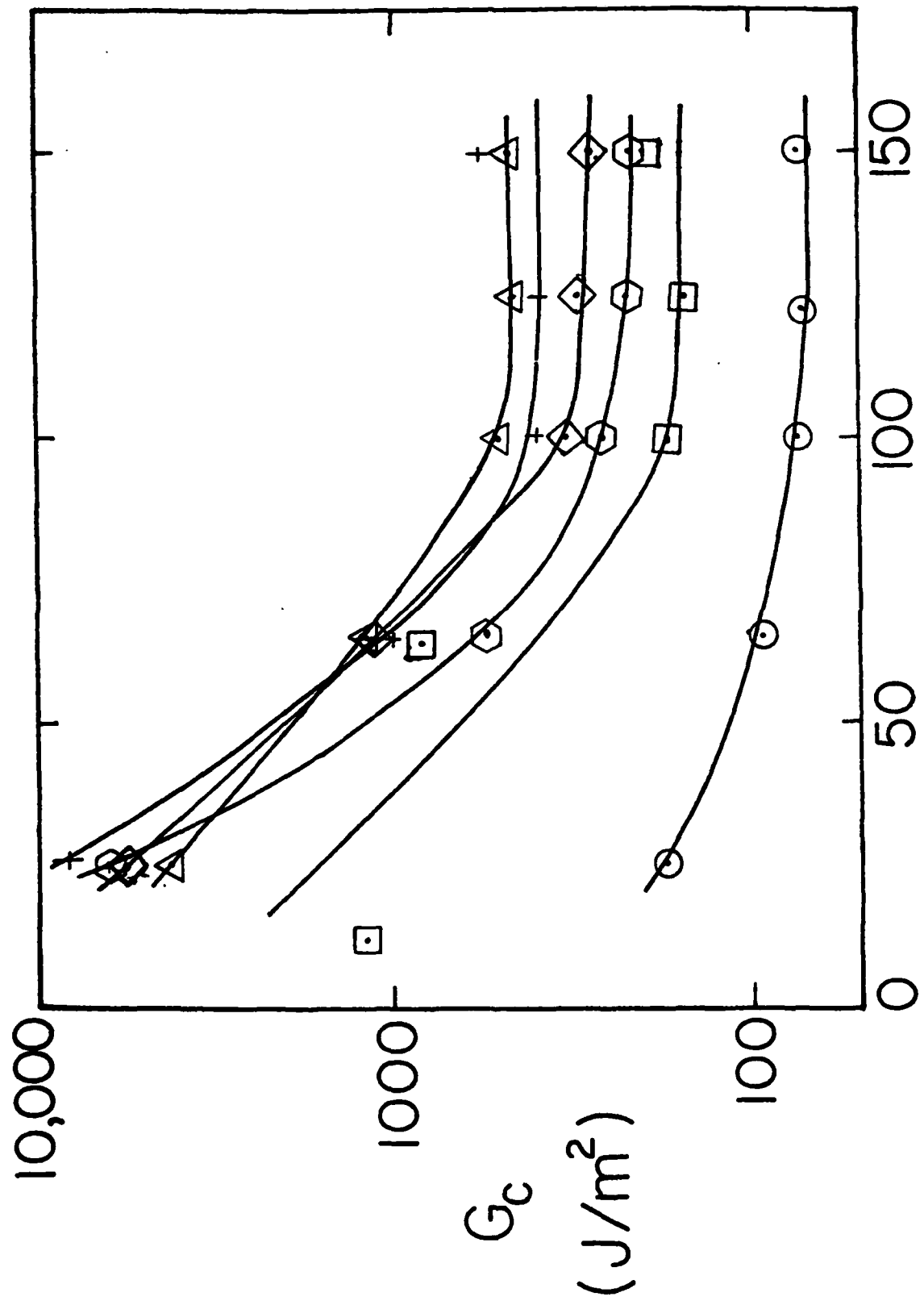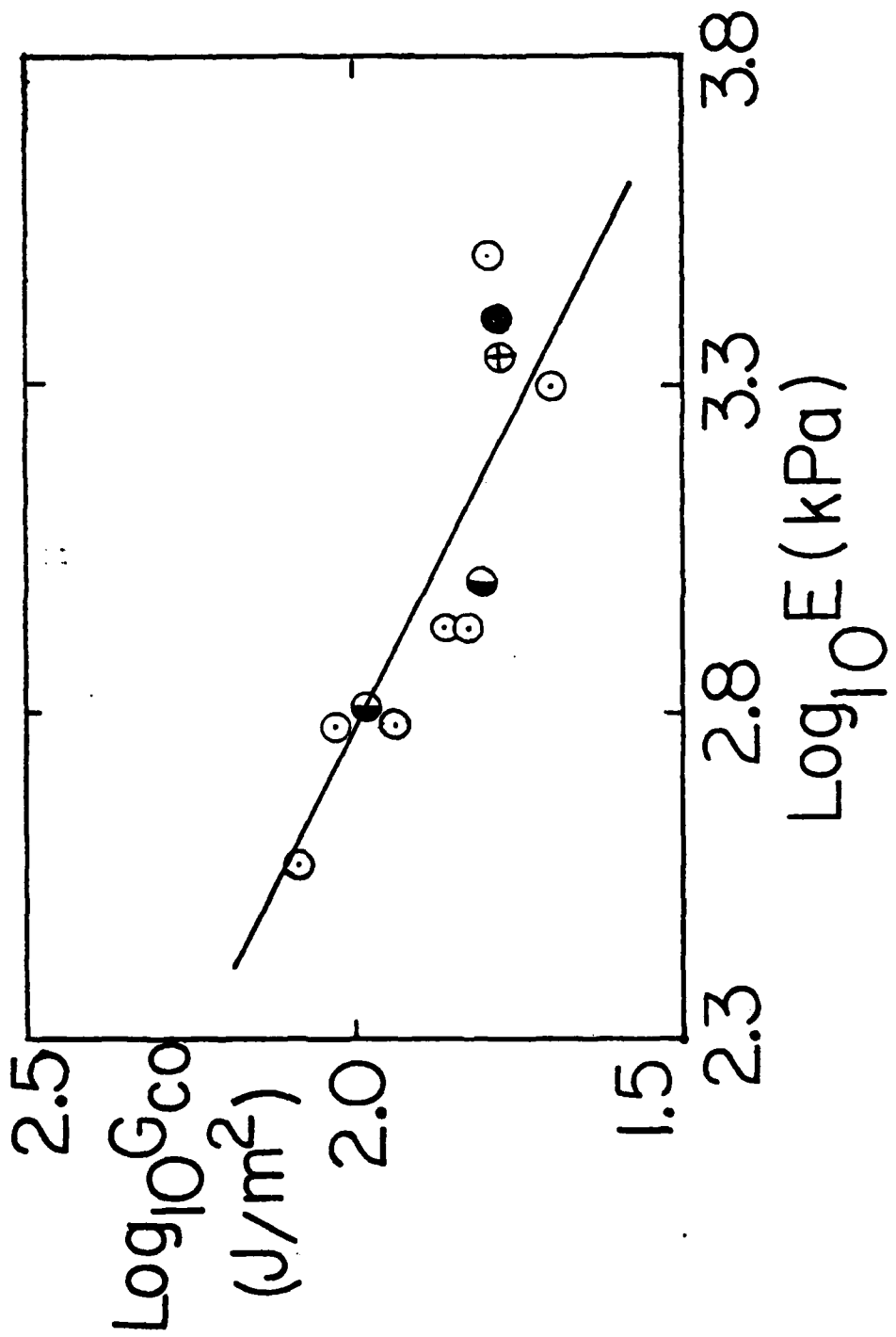
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