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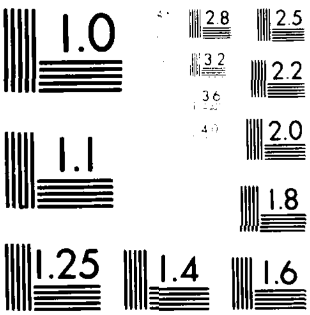

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by

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20. ABSTRACT (*Continue on reverse side if necessary and identify by block number*)

The tear strength of polydimethylsiloxane (PDMS) networks was found to be only about one-third as large as that of polybutadiene (PB) or polyisoprene (PI) networks of similar M_c when the tear strength was measured under threshold conditions, i.e., at high temperatures, low rates of tearing, and with swollen samples. This striking difference in strength is attributed to the smaller length and extensibility of PDMS molecules in comparison with PB or PI molecules of the same molecular weight. Networks formed by trifunctional or tetrafunctional endlinking reactions with difunctional PDMS polymers were found to be

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SECURITY CLASSIFICATION OF THIS PAGE (*When Data Entered*) only slightly stronger under threshold conditions, by up to 30 per cent, than PDMS networks formed by random crosslinking to the same M_c. Endlinked PB networks were found to have substantially the same threshold strength as randomly-linked PB networks of the same M_c. Thus, the threshold tear strength does not appear to depend strongly upon the uniformity of network strand lengths.

THRESHOLD TEAR STRENGTH OF SOME MOLECULAR NETWORKS

by

A. N. Gent and R. H. Tobias

Introduction

When dissipative processes are minimized, the tear strength of elastomeric materials is found to reach a lower limit, termed here the threshold strength (1,2). Experimentally, the threshold strength is reached at high temperatures, at low rates of tearing and when the material is highly swollen with a low-viscosity liquid. Its magnitude has been predicted theoretically from the length of the molecular strands comprising the network and the dissociation energy of the chemical bonds comprising the strand (3). Expressed as the energy T_o required to tear through a unit area of the material, the theoretical result takes the form

$$T_o = KM_c^{\frac{1}{2}} \qquad (1)$$

where M_c is the mean molecular weight of the network strands and K is a constant involving the mass, length and effective flexibility of a monomeric unit, the density of the polymer and the dissociation energy of the relevant bonds. For C-C molecular strands K is predicted to be about 0.3 $J/m^2/(\text{molecular weight unit})^{\frac{1}{2}}$. Experimental values of T_o for randomly-crosslinked networks of polybutadiene were found to be consistent with equation 1, when K was given a somewhat higher value, about 1.0 $J/m^2/(\text{molecular weight unit})^{\frac{1}{2}}$. Thus, apart

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from this numerical discrepancy, the threshold strength of polybutadiene networks is reasonably well accounted for. We now address two further aspects of threshold strength. Does the tear strength of other elastomers, of different chemical type, conform to equation 1? And are randomly-crosslinked materials weaker than more regular networks, prepared, for example, by linking strands of uniform molecular weight into a network by endgroup coupling reactions? A higher tensile strength has been reported for endlinked networks of polyisoprene in comparison with randomly-crosslinked networks of similar average strand length (4). However, those measurements were not made under threshold conditions and did not examine a wide range of strand lengths, so that direct comparison with molecular theory is not possible.

Measurements have now been carried out on endlinked and randomly-linked samples of polybutadiene, polydimethylsiloxane, and randomly-linked samples of cis-polyisoprene. The results are presented here.

Experimental Details

(i) Endlinked PDMS

Linear polydimethylsiloxanes with vinyl endgroups were supplied by Dow Corning Corporation. Three different molecular weight ranges were employed. Membrane osmometry yielded values for $\bar{M}_n$ of 16,000 24,000, and 37,000 g/g-mole. Endgroup analysis using mercuric acetate (5) gave vinyl contents of 0.47 ± 0.03, 0.24 ± 0.02, and

0.15 ± 0.02 per cent, corresponding to values for M_n of 11,500, 22,500 and 36,000 g/g-mole. GPC data gave $\bar{M}_w/\bar{M}_n$ ratios of approximately 2.0, as reported by Valles and Macosko (6,7) for their similar polymers.

Trifunctional and tetrafunctional silane linking agents were supplied to us by Prof. Macosko. They consisted of trikis-dimethylsiloxyphenylsilane and tetrakis-dimethylsiloxysilane and are denoted here A3 and A4, respectively. Gas chromatography, carried out by Prof. Macosko, revealed that they were approximately 95 and 89 per cent pure. Si-H group analysis (5) gave average functionalities of 3.15 and 3.50, somewhat different from the expected values of 3 and 4, indicating that other constituents are present.

The linking agent, A3 or A4, was mixed in various concentrations with the divinyl-PDMS, together with 5 ppm of a Pt catalyst (8). The mixture was then degassed and cast as a thin sheet on a Teflon surface. Complete reaction was found to occur on heating for about 3 days at 70°C, as judged by equilibrium swelling measurements in benzene; thereafter, 4 days at 70°C was used to ensure complete reaction.

As shown in Figure 1, values of M_c calculated from equilibrium swelling ratios in benzene (9), were found to depend strongly upon the concentration of endgroups in the linking agent relative to those in the polymer. The highest effective degree of crosslinking, i.e., the lowest degree of swelling and the lowest value for M_c, was obtained at a characteristic endgroup ratio lying between about 1.1 and 1.6 instead of the expected ratio of 1.0. Thus, even when allowance is made for the true functionality of the linking agent, it is still necessary to employ a greater amount than expected to produce a minimum value for M_c. Presumably, a significant fraction of polymer endgroups do not become linked into the network until an excess of linking agent is present. This implies that the junction points are not exclusively trifunctional or tetrafunctional in nature.

Networks were prepared in all cases using the amount of endlinking agent necessary to give a minimum M_c. Values of M_c were calculated from the Mooney-Rivlin elasticity coefficient C_1, determined from tensile stress-strain measurements (10),

$$M_c = \rho RT/2C_1, \tag{2}$$

where ρ is the density, 0.97 g/ml, R is the gas constant and T is absolute temperature, for comparison with earlier work (2).

(ii) Randomly - crosslinked PDMS

The polydimethylsiloxane (PDMS) used to make random networks was obtained from General Electric. Membrane osmometry showed $\bar{M}_n$ to be 430,000 g/g-mole. The polymer was mixed with various amounts of a free-radical crosslinking agent, dicumylperoxide (Di-Cup R, Hercules Chemical Co.). Samples were then pressed into sheets and crosslinking was effected by heating for 2 h at 150°C in a heated press. M_c values were calculated using equation 2.

(iii)Endlinked PB

Endlinked polybutadiene samples were provided by Prof. F. N. Kelley and Mr. Long-Ji Su of these laboratories. They were made by reacting toluene -2,4- diisocyanate with the vinyl endgroups of vinyl-terminated polybutadiene (Arco R45-HT) and then employing trimethylol-propane as a trifunctional linking agent. The prepolymer was also chain-extended with 1,4 - butanediol to give a higher M_c value on subsequent endlinking. M_c values, calculated by means of equation 2, were 3,100 g/g-mole for the sample made with the initial polymer and 7,100 g/g-mole when the chain-extended material was used.

(iv) Randomly - crosslinked PB and PI

Polybutadiene (Diene 35 NFA, Firestone Tire and Rubber Co.) and cis-polyisoprene (Natsyn 2200, Goodyear Tire and Rubber Co.) were crosslinked with dicymylperoxide, as for PDMS. M_c values were also calculated by means of equation 2.

(v) Measurements of threshold tear strength

Rectangular strips, about 60 mm long, 10 mm wide and 1.4 mm thick were scored along a central line to a depth of about 0.7 mm, leaving about one-half of the original thickness to be torn through. The tear energy T was calculated from the measured tear force F,

$$T = 2\ F/t, \tag{3}$$

where t is the width of the tear path (Figure 2). Tearing was found to take place at an angle of approximately 45° to the sheet thickness (11).

Measurements were carried out on both swollen and unswollen samples, using m-xylene or silicone oil as swelling liquids with PDMS networks and m-xylene or paraffin oil with PB and PI networks. Samples were torn at temperatures between 70°C and 140°C. Values of the tear energy T for swollen samples were multiplied by λ_s^2, where λ_s is the linear swelling ratio, to take into account the reduced number of network strands crossing the tear path (1-3). Close agreement was obtained with unswollen samples at the high temperatures and low rates of tearing (about 4μm/s) used in the present experiments. The mean values have therefore been taken as measures of the threshold tear strength T_o.

Experimental Results and Discussion

Experimentally-determined values of T_o for PDMS networks are plotted in Figure 3 against values of M_c calculated from the elastic coefficient C_1 by means of equation 2. T_o was found to be accurately proportional to $M_c^{½}$, in accordance with equation (1), with the coefficient of proportionality K being about 0.30, 0.25, and 0.23 $J/m^2/$(molecular weight unit)^{½} for the A4, A3, and randomly-linked materials, respectively. These differences are small, barely significant, but in the expected direction. Values of T_o are also shown in Figure 3 for the other materials examined. Again, a proportionality to $M_c^{½}$ was found, in accordance with theory. Moreover, the present values for endlinked PB and randomly-linked PI are in good agreement with previously-reported data on randomly-linked PB, with $K = 0.85\ J/m^2$. This is much larger than for the PDMS materials, however. Thus, at the same value of M_c, elastomeric networks based on C-C molecular chains have a threshold strength about three times that of Si-O networks.

Although the bond dissociation energies for C-C and Si-O bonds are quite similar, 89 and 80 kcal/g-mole respectively, the molecular weight per main-chain atom is considerably higher for PDMS (37 molecular weight units) than for PI (17) and PB (13.5). The extended length of a network strand is therefore much smaller for PDMS at the same value of M_c. There are also steric restrictions on straightening the Si-O chain due to unequal main-chain bond angles. Thus, the threshold strength of

PDMS networks would be expected to be less than one-half as large as for C-C networks, in accordance with observation.

Conclusions

The threshold tear strength of elastomeric molecular networks does not appear to depend strongly, if at all, upon the uniformity of network strand lengths. It is found to be proportional to $M_c^{1/2}$ where M_c is the mean molecular weight of the strands, in accordance with the theory of Lake and Thomas (3). However, it is considerably smaller for Si-O networks than for C-C networks at equal M_c values. This is attributed to differences in strand length and extensibility.

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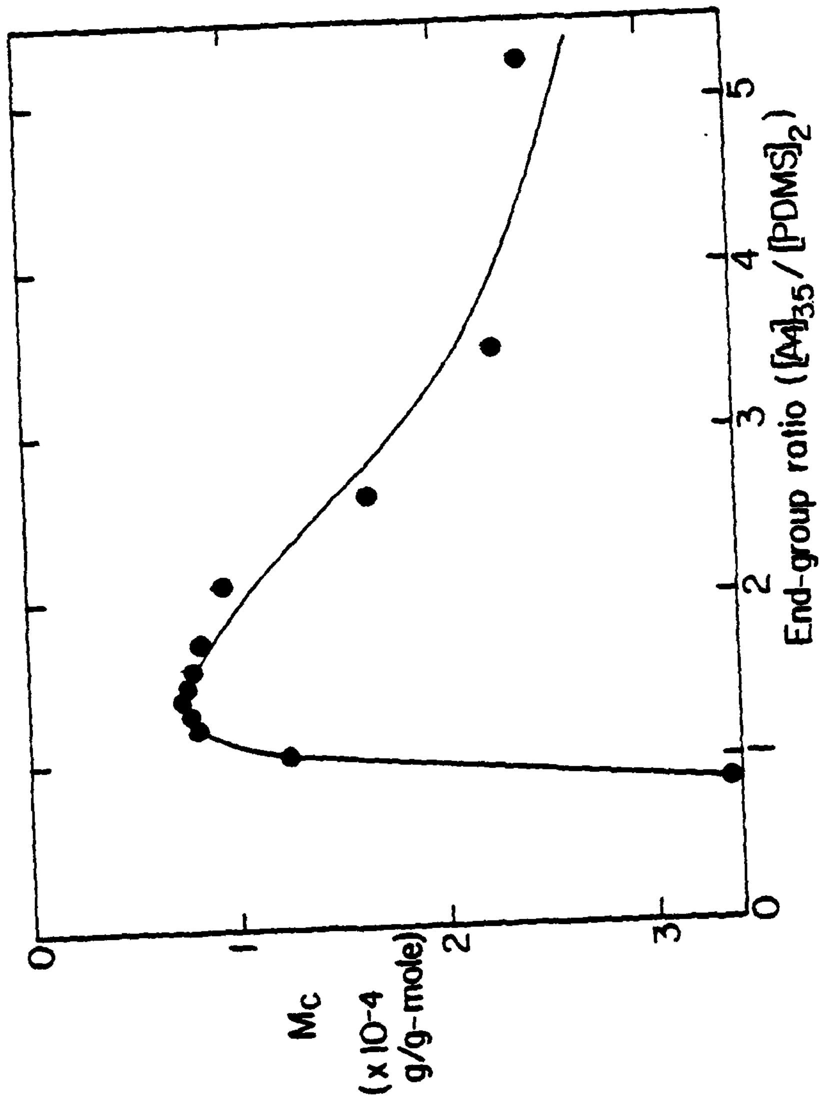
Figure Legends

Figure 1. M_c from equilibrium swelling in benzene *vs* concentration of A4 endlinking agent in divinyl-PDMS, M_n = 11,500.

Figure 2. (a) Tear test (b) Sketch of torn cross-section.

Figure 3. Threshold tear energy T_o for PDMS networks (○,△,□), PB networks (●,▲), and PI networks (◐), *vs* molecular weight M_c between crosslinks calculated from C_1. Random crosslinking: ○, ◐, ●; trifunctional endlinking: △, ▲; tetrafunctional endlinking: □ .

Figure 1.



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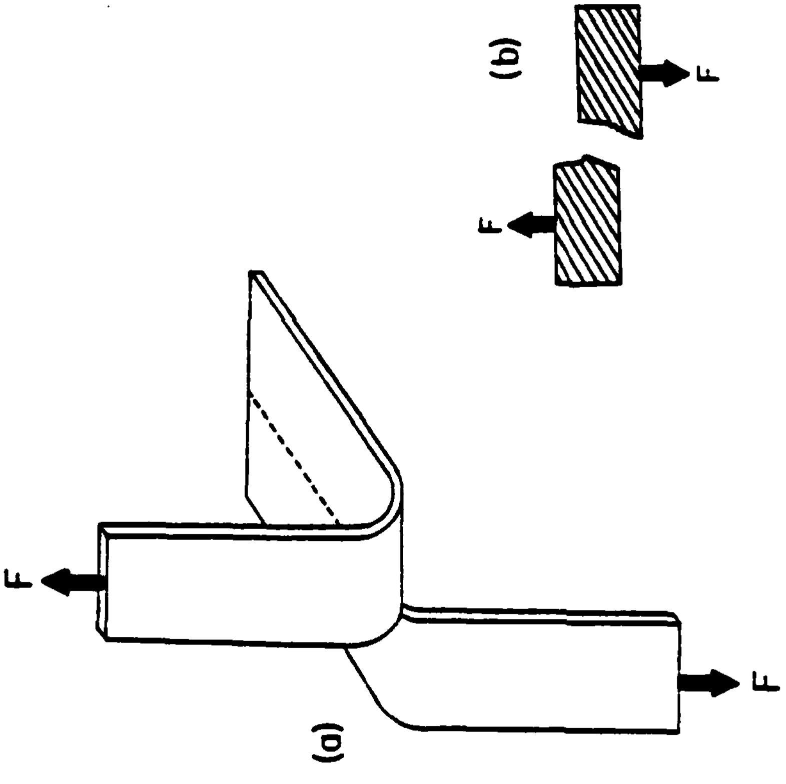

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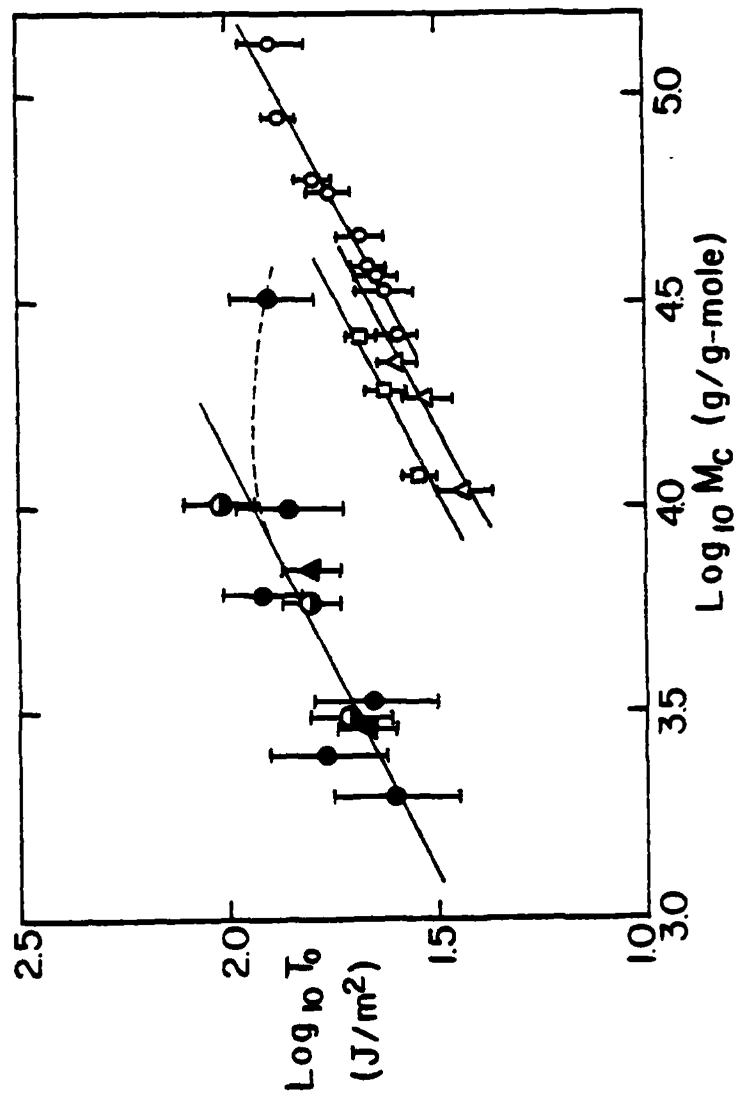

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