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EFFECT OF INTERFACIAL BONDING ON THE STRENGTH OF ADHESION
OF ELASTOMERS II. DISSIMILAR ADHERENDS

by

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sheet of the same elastomer and the crosslinking then taken to completion, the strength of adhesion under threshold conditions was found to be qualitatively in accord with the predictions of a simple theoretical treatment for the degree of interlinking in terms of the corresponding homogeneous crosslinking reaction. Whereas the theory suggests that the effective degree of interlinking will be one-half of that generated in a homogeneous system, the experimental results were in accord with a figure of about 70 per cent. When a layer of one elastomer was bonded to a layer of the other in a similar way, the strength of adhesion was found to be relatively high when the initial fully-crosslinked layer was BR and relatively low when it was EPR. These results were also in qualitative agreement with theoretical predictions for the degree of chemical interlinking developed between layers differing in chemical reactivity. Thus a general correlation appears to hold between the threshold strength of adhesion and the amount of interlinking.

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Introduction

A previous study was concerned with the self-adhesion of elastomer layers bonded together by covalent crosslinks (1). Two identical partially-crosslinked elastomer sheets were brought together and chemically interlinked by taking the crosslinking process to completion. The degree of interlinking was inferred from the increase in crosslink density of the two layers during the second stage of crosslinking, carried out while the layers were in intimate contact. For these symmetrical specimens a direct proportionality was found to hold between the threshold strength of adhesion, i.e., the work of separation per unit of interfacial area measured at low rates of separation and at high temperatures, and the inferred degree of interfacial interlinking.

Experiments have now been carried out on unsymmetrical specimens, in which the two elastomer layers to be bonded together differ either in initial degree of crosslinking or in chemical reactivity. The results are reported here and compared with those obtained previously for symmetrical specimens prepared from the same elastomers.

Experimental Details

Two types of unsymmetrical joint were prepared. The first consisted of a fully-crosslinked sheet of one

elastomer bonded to a partially-crosslinked sheet of the same elastomer by pressing them together and completing the crosslinking process. This procedure is shown schematically in Figure 1a. A method for estimating the amount of interlinking of the two sheets is outlined in the following section of the paper.

The second type of unsymmetrical joint is shown schematically in Figure 1b. In this case different elastomers were employed for the two sheets. Thus, a fully crosslinked sheet of one elastomer; for example, polybutadiene (BR); was bonded to a partially-crosslinked sheet of another elastomer; for example, an ethylene-propylene copolymer (EPR); by pressing them together and completing the crosslinking process. Again, a method for estimating the amount of interlinking developed is presented in the following section.

The elastomers used for preparing test samples were the same as before: an anionically-polymerized polybutadiene (Diene 35 NFA, Firestone Rubber and Latex Company), and an ethylene-propylene copolymer (Vistalon 404, EXXON Chemical Company). They were crosslinked with a free-radical crosslinking agent, dicumyl peroxide, using recipes given previously (1). Flat sheets, about 0.5 mm thick, were prepared by heating for a time t_1 in a heated press at 150°C. The initial degree of crosslinking depended upon the time t_1 . It was determined by measuring the equilibrium degree of swelling of the partially-crosslinked sheets in n-heptane

(BR) or in benzene (EPR), as described before (1). It was characterized by the number $\underline{\nu}$ of molecular network strands per unit volume, calculated by means of the Flory-Huggins relationship (2).

Peeling experiments were employed to determine the work \underline{G}_a of separation per unit area of bonded interface. These measurements were made at a relatively high temperature, 100°C, and at a low rate of peel, 0.4 $\mu\text{m/s}$ so that the values obtained will be close to the threshold strengths of adhesion when contributions from viscoelastic effects in the peeling layers are absent (1). They have been taken here as the threshold strengths \underline{G}_0 .

Estimation of the amount of interfacial bonding

(i) Different degrees of initial crosslinking.

Let us assume that one layer has been crosslinked fully, so that all of the crosslinking agent withⁱⁿ it has been used up. The corresponding density of network strands is denoted $\underline{\nu}_f$. The other layer has been partially crosslinked initially and has a strand density of $\underline{\nu}_i$ before the two layers are brought into contact.

It is now assumed that some diffusion of the unreacted crosslinking agent takes place while the two sheets are being crosslinked in contact. As a result of this diffusion, the contacting region of the^{initially} fully-crosslinked layer undergoes additional crosslinking by an amount $\underline{\nu}_e$, so that it reaches a final degree of crosslinking of $\underline{\nu}_f + \underline{\nu}_e$.

Meanwhile the other layer will crosslink to a lesser extent than it would have otherwise done, because of the loss of crosslinking agent. It will, therefore, reach a final level of crosslinking of $\underline{v_f - v_e}$ in the region near the interface.

Thus, the interface will separate two regions of elastomer that were initially crosslinked to levels $\underline{v_f}$ and $\underline{v_1}$, and are ultimately crosslinked to levels $\underline{v_f + v_e}$ and $\underline{v_f - v_e}$. They have, therefore, undergone different amounts of additional crosslinking whilst in contact; $\underline{v_e}$ and $\underline{v_f - v_1 - v_e}$, respectively.

The degree of interlinking of the two layers is best represented by the mean of these two values, i.e., by the average extent of additional crosslinking in the two contacting layers. Thus,

$$\overline{\Delta v} = (v_f - v_1)/2. \quad (1)$$

It should be noted that this is one-half of the interlinking developed in a symmetrical system in which each elastomer layer is initially crosslinked to a level $\underline{v_1}$.

(ii) Different elastomer layers.

Again, it is assumed that one layer has been fully crosslinked initially. The initial levels of crosslinking are thus represented by $\underline{v_{f_2}}$, corresponding to the equilibrium value of \underline{v} for elastomer 2, and $\underline{v_1}$, the initial value for elastomer 1. As a result of diffusion of the crosslinking agent from the first layer into the second during the interlinking stage, the final level of crosslinking

attained by elastomer 1 will be lower than if it were crosslinked alone. It is denoted $\underline{v_{f_1}} - \underline{v_e}$, where $\underline{v_e}$ denotes the density of network chains that are lost to elastomer 1 in the vicinity of the interface because of migration of crosslinking agent into elastomer 2.

In the present instance it is necessary to take into account possible differences in efficiency of crosslinking of the two elastomers. The amount of crosslinking agent that creates $\underline{v_e}$ crosslinks in elastomer 1 will create a different number, say $\alpha \underline{v_e}$ crosslinks, in elastomer 2, where α denotes the relative efficiency of crosslinking in elastomer 2 compared to elastomer 1. Then the final level of crosslinking for elastomer 2 becomes $\underline{v_{f_2}} + \alpha \underline{v_e}$.

The two layers have ^{now} undergone the following changes in crosslinking whilst in contact: $\underline{v_{f_1}} - \underline{v_1} - \underline{v_e}$ and $\alpha \underline{v_e}$. The mean value $\underline{\Delta v}$ is, therefore, given by

$$2 \underline{\Delta v} = (\underline{v_{f_1}} - \underline{v_1}) + (\alpha - 1) \underline{v_e}. \quad (2)$$

$\underline{\Delta v}$ provides an estimate of the degree of interlinking. It can be greater or less than the amount of interlinking developed with layers of the same elastomer, given by equation (1), depending upon the value of $(\alpha - 1)$. If α is greater than 1, then the degree of interlinking is enhanced, whereas, if α is less than 1, the degree of interlinking is reduced, in comparison with layers of

the same elastomer subjected to the same bonding conditions.

The efficiencies of crosslinking for BR and EPR are apparently quite different. We note that whereas about 0.1 - 0.2 per cent of dicumyl peroxide is sufficient to crosslink BR adequately, 2.7 per cent is required to crosslink EPR to a similar degree. Thus, if a layer of BR represents elastomer 1, i.e., is fully-crosslinked initially, and EPR represents elastomer 2, then the parameter α may be estimated to be in the range 13 - 27, much greater than unity. On the other hand, if an EPR layer is fully-crosslinked initially, and is then bonded to a partially-crosslinked layer of BR, then the corresponding value of α becomes $1/13 - 1/27$, much smaller than unity. In the former case a higher degree of interlinking would be expected from equation (2), and in the latter case a much lower degree of interlinking, than for layers of the same elastomer. These conclusions have been examined experimentally, as described in the following section.

Measurements of the strength of adhesion

(i) Bonding a partially-crosslinked BR layer to a fully-crosslinked BR layer.

Experimentally-determined values of the work G_0 of separation per unit area of bonded interface, are plotted in Figure 2 against the degree of interlinking

$\Delta v = v_f - v_1$ that would have occurred in a symmetrical system of two partially-crosslinked BR layers.

Experimental results obtained previously with symmetrical specimens are plotted as a full line in Figure 2 for comparison. The present values for G_o , represented by open circles in the figure, are seen to be approximately proportional to Δv but considerably smaller than before, corresponding to degrees of interlinking of about 70% of Δv . Equation (1) predicts that the effective interlinking would be 50% of Δv . Thus, the experimentally-measured strengths of adhesion are qualitatively in accord with the theoretical predictions, but somewhat higher than expected.

(ii) Bonding a partially-crosslinked EPR layer to a fully-crosslinked EPR layer.

Similar experiments were carried out with EPR layers. The results for G_o are represented in Figure 3 by open circles. They are compared there with results obtained previously for symmetrical EPR specimens. Again, values of the work G_o of separation are roughly proportional to the degree of interlinking Δv for corresponding symmetrical specimens, but smaller, as if the actual interlinking was only about 70% of Δv . Thus, as for BR layers, the experimental results for G_o are qualitatively in accord with the simple theory given in section 3 but somewhat larger.

(iii) Bonding an EPR layer to a BR layer, and vice versa.

Experiments were first carried out with fully-crosslinked sheets of EPR that were pressed into contact with partially-crosslinked sheets of BR and then the crosslinking was taken to completion. Measured values of the detachment energy G_0 are plotted as triangles in Figure 2 against the degree of interlinking Δv that would have occurred using two partially-crosslinked sheets of BR. The strength of adhesion increased linearly with the degree of interlinking Δv but at any level of interlinking the values of G_0 were much smaller than for a symmetrical testpiece with two BR layers. They corresponded roughly to levels of interlinking of only about 40% of Δv , or about one-half of the value of Δv inferred when a BR layer was bonded to a fully-crosslinked BR layer. In the EPR/BR system the parameter α , representing the relative efficiencies of crosslinking in EPR and BR, is quite small, certainly less than unity and probably less than 0.1. Equation (2) then predicts a low level of interlinking in comparison with the two BR layers. Thus, the experimental results are in good qualitative agreement with theoretical predictions.

When a partially-crosslinked layer of EPR was joined to a fully-crosslinked layer of BR by completing the crosslinking reaction with the two layers in contact, then the strength of adhesion was found to be relatively high. Values of G_0 are plotted in Figure 3 against the degree of interlinking that would have occurred with two partially-crosslinked EPR layers. The present experimental results, represented by triangles, are seen to be quite close to the linear relation obtained previously for symmetrical specimens made by joining two partially-crosslinked EPR layers. Thus, the degree of chemical interlinking may be inferred to be similar also. This high level of interlinking is also consistent with equation (2), recognizing that the parameter α , denoting the relative crosslinking efficiencies of the two elastomers, is now much greater than unity. In both cases, therefore, the measured strengths of adhesion for dissimilar elastomers are in good qualitative agreement with the predictions of a simple theory for the degree of chemical interlinking. The threshold strength appears to be directly proportional to the degree of interlinking.

Discussion and conclusions

The experimental systems used in this work represent models for the covulcanization of incompatible elastomers, either in the form of compounds when the elastomers are mixed together in a finely-divided state, or in structures where two elastomer layers are plied together (as in tire manufacture). They might also be regarded as general models of thermosetting adhesives when some reaction takes place across the interface during gelation or setting. In all of the cases studied, whether the layers to be joined together consisted of the same elastomer crosslinked to different degrees or of different elastomers with different reactivity, a general proportionality appears to hold between the mechanical strength of the joint, determined under threshold conditions, and the amount of chemical interlinking existing between the adhering layers. For the simple systems examined here, a method has been proposed for estimating the degree of interlinking from the course of the homogeneous crosslinking or gelation process within an isolated layer. This theory appears to be qualitatively correct, predicting the overall character of the results and the approximate magnitudes. Indeed, in view of the approximations made in developing the theory, the general level of agreement for widely-different systems can be regarded as quite satisfactory.

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Figure Legends

Figure 1. Examples of interlinking reactions between dissimilar adherends.

- (a) A partially-crosslinked sheet of EPR is crosslinked to completion in contact with a fully-crosslinked sheet of EPR.
- (b) A partially-crosslinked sheet of EPR is crosslinked to completion in contact with a fully-crosslinked sheet of BR.

Figure 2. Detachment energy G_0 for partially-crosslinked BR layers subsequently interlinked:

- (a) to each other (full curve, taken from reference 1),
- (b) to fully-crosslinked BR layers (O),
- (c) to fully-crosslinked EPR layers (Δ).

Figure 3. Detachment energy G_0 for partially-crosslinked EPR layers subsequently interlinked:

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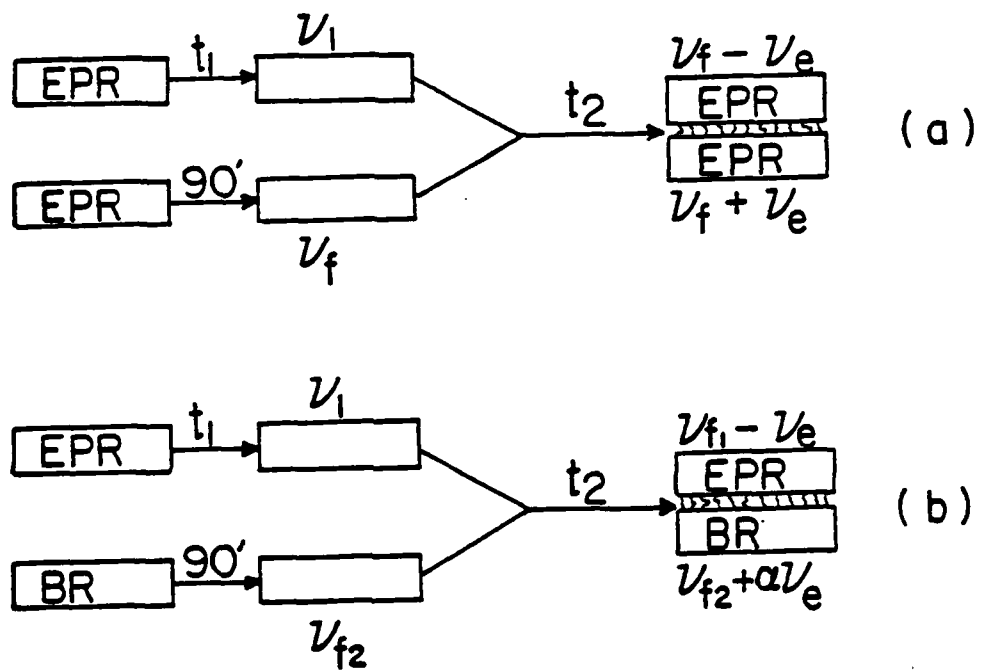
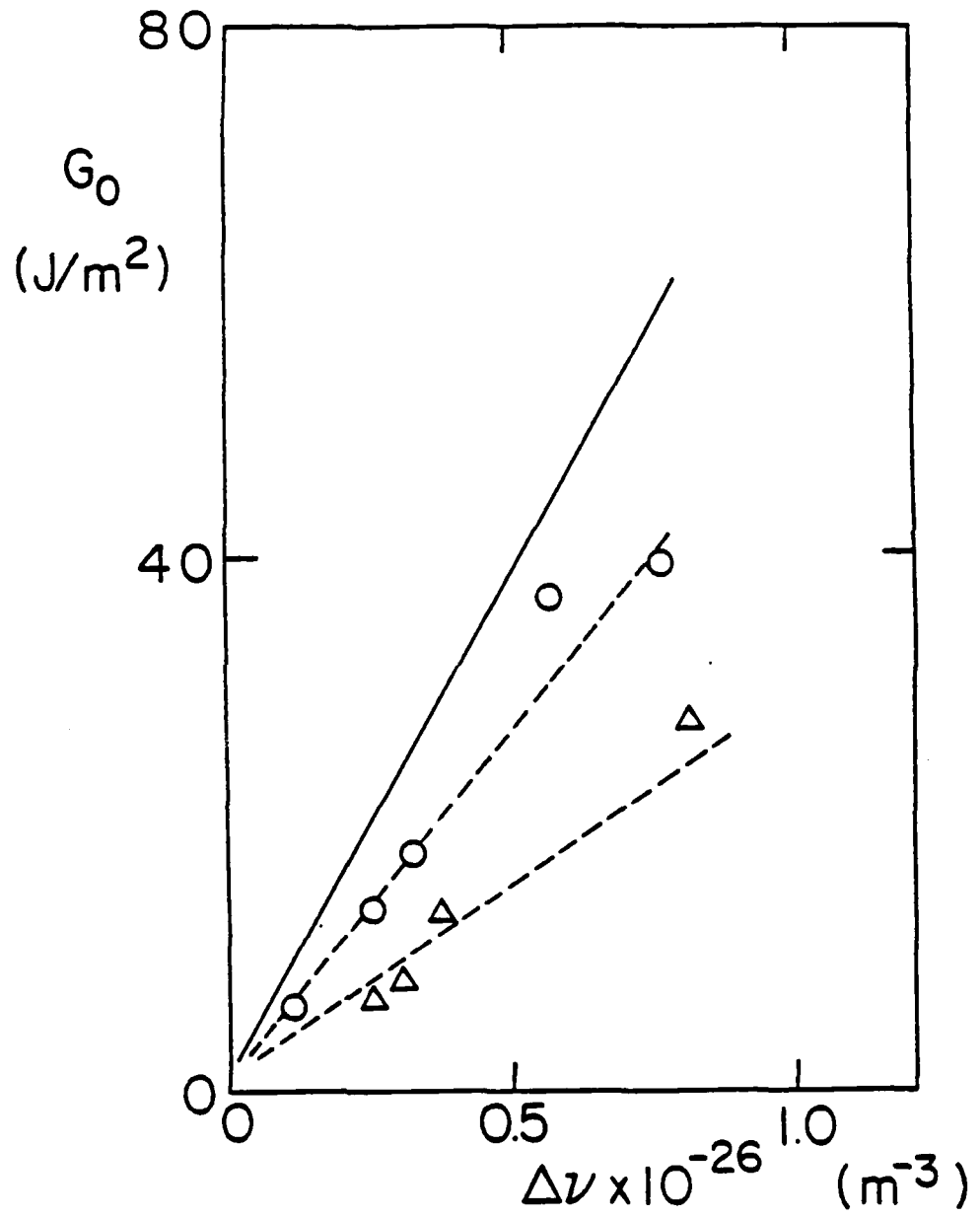


FIGURE 1

FIGURE 2

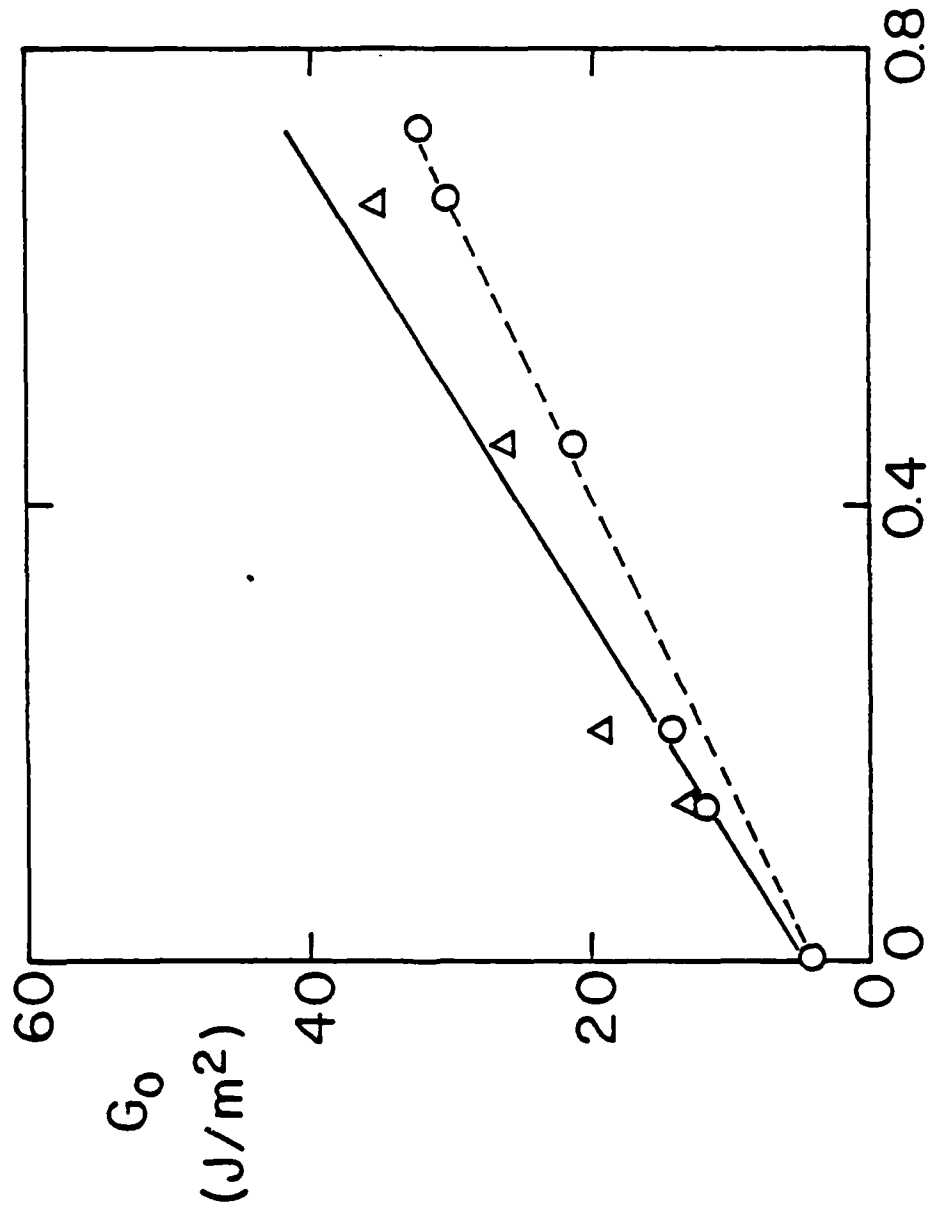


FIGURE 3

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