

Kinetic Analysis of the Autohesion of Raw SBR Samples

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Synopsis

A kinetic analysis of the autohesion of raw, cold, emulsion SBR rubbers having number-average molecular weights ranging from 74,000 to 169,000 was undertaken by using the modified Wallace tackmeter. The autohesion was observed to be a reversible process following first-order kinetics with respect to the formation of elementary area bonds. The application of greater contact pressures during autohesion causes an increase in the kinetic work, bond strength, and time required for bond rupture. A practical aspect of the study shows that the contact time necessary to form a complete area bond is reduced by: (1) the annealing of samples to release internal stresses; (2) production of clean and very smooth surfaces; (3) equilibrating samples under pressure prior to joining them; (4) lowering the sample temperature. The raw SBR offering the best potential autohesive properties has a \bar{M}_n of 133,000 and ML-4 (212°F.) of 66.

INTRODUCTION

The increasing importance of the industrial uses of synthetic elastomers has prompted applied scientists to study in greater detail the chemical and physical properties of raw rubbers. Their wide acceptance today precludes any attempt to give a complete list of applications. The proper selection of an elastomer for a recipe, from the numerous synthetic polymers available, permits the production of unique structural bodies which depend upon both good molding and bonding properties in the unvulcanized state. In this respect, autohesion studies have been important in reporting the comparative bonding properties of many new elastomers. Excellent reviews of the problems related to autohesion and adhesion have been recently published by Voyutskii,¹ Weidner and Crocker,² and Bussemaker.³

In this research, the autohesive properties of polymers are viewed with a kinetic interpretation which introduces the variables of time, temperature, and pressure. The basis for the development of the ideas presented here was taken from a paper published by Furukawa.⁴ The extension of these ideas to the effects of contact pressure and rate of bond development provide a more comprehensive analysis of autohesion.

In general, the two most important criteria in this study of autohesion are the rate of development of the elementary area bond and the energy required for its rupture. Ideally, the polymer exhibiting excellent auto-

hesion characteristics should form the area bond instantaneously upon contact and require large energies for bond rupture. The inability of a polymer to meet one or both of these conditions will cause inferior autohesive properties.

It is the object of this investigation to compare the autohesive properties of five cold-emulsion, raw SBR samples with respect to variations in the number-average molecular weights. The results of osmometric and rheological studies of these polymers are presented in Table I. The polymers were prepared with the same redox catalyst system and have a bound styrene content of 23.5 wt.-%.

TABLE I
Information on the Raw SBR Samples

Sample number	Sample type ^a	ML-4 (212°F.)	Intrinsic viscosity (η), 30°C. in toluene	Solubility in ethanol-toluene azeotrope, % ^b	10% solution viscosity at 25°C.	Number-average molecular weight $\times 10^{-5}$
160	ASRC 3105	36	1.72	MA 6.5	222	0.74
161	1551	56	1.98	RA 7.8	(520)	1.07
162	1-OST	(66)	2.32	MA 7.2	1075	1.33
163	1712	(91)	2.96	MA 6.4	3675	1.44
164	8214	(95)	3.14	MA 3.7	6300	1.69

^a Microstructure: 65-70% *trans*, 17-20% vinyl, 10-17% *cis*.

^b MA = mixed acid; RA = rosin acid.

KINETIC ANALYSIS OF THE AUTOHESIVE AREA BOND

A description of the tackmeter and the procedure for determining autohesion has been reported by Skewis.⁵ The samples were molded from raw latex crumb at 100-125°C. in Teflon-coated molds under a hydraulic platen pressure of 5 tons on a 5-in. ram, 4000-lb. tube. The molding operation produced rectangular strips of raw rubber 6 in. long, $\frac{3}{4}$ in. wide at the base, $\frac{1}{2}$ in. wide at the contact surface, and $\frac{3}{8}$ in. in thickness. These were cut into 1-in. lengths for the autohesion experiments.

Since the molding operation introduced stresses into the samples, the samples were annealed in their molds at 50-70°C. for $\frac{1}{2}$ hr. under the pressure of the mold cover (0.066 psi). If the samples began to deform by twisting in the mold, the annealing treatment was repeated.

The relaxation of internal stresses considerably increased the autohesive strength of the raw SBR samples. The surface smoothness was improved by covering the contact surface with aluminum foil and warming the samples to 50°C. for 2 min. The aluminum foil was stripped after cooling to room temperature. This procedure was repeated until the raw rubber surface had a glassy appearance. The sample surfaces were protected by the aluminum foil covering, which was removed immediately before an

autohesion measurement. The surfaces were exposed to the air for approximately 15 sec. prior to joining them.

Briefly, the tackmeter experiment involves the contacting of two molded samples for a known contact pressure and contact time which is followed by measuring the time to separate them under a given breaking pressure at constant temperature. These experiments are divided into two categories: (1) the use of an instantaneous contact pressure or non-equilibrium conditions; (2) the equilibrating of the samples under the contact pressure to be used for long intervals of time (2 weeks) prior to the autohesion experiment or equilibrium conditions.

Equilibrium Conditions

Under equilibrium conditions, the elastomer reaches a steady state with respect to the contact pressure prior to the autohesion experiments. The sample is kept under a constant pressure for at least 7 days either in a Teflon-coated mold or the two elastomer surfaces are pressed against each other, aluminum foil being used to separate them in the tackmeter. In this manner, the free volume to be attained is available for the autohesion at the beginning of the surface contact.

The autohesion process is viewed as an interdiffusion of polymer segments through a network of micropores. The ultimate crosslinking is due to the interaction of van der Waals forces between segments and not a chemical type bonding as in the vulcanization of rubber.

By defining the time required for rupture, t_B , as the number Z_0 of bonds per square centimeter divided by the rate of disappearance of the crosslinks, $-dz/dt$, and applying a modification of Eyring's rate theory for large breaking forces, it is possible to derive an expression for the process of breaking a bond:

$$t_B = Z_0/(-dz/dt) \quad (1)$$

$$-dz/dt = k_B e^{w/kT} \quad (2)$$

$$\ln t_B = \ln(Z_0/k_B) - (W/kT) \quad (3)$$

$$W = F_B a^2 (N_0)^{2/3} a^*/2 A_S Z_0 = F_B a^* 2.022 A_S Z_0 \quad (4)$$

$$F_B = -K \ln t_B + K \ln (Z_0/k_B)_{F_B=0} \quad (5)$$

where W is the work required for rupture of a crosslinked segment (ergs), k_B is the rate constant for bond rupture ($\text{sec.}^{-1} \text{ cm.}^{-2}$), F_B is the breaking force (dynes), A_S is sample area = 1.613 cm.^2 , a^2 is the area of a carbon-carbon segment (square centimeters), $(N_0)^{2/3}$ is the number of carbon-carbon segments per square centimeter assuming a void volume of 26%, $a^*/2$ is the distance (centimeters) moved by a crosslinked segment to cause bond rupture, $K = 2.022 A_S Z_0 kT/a^*$, k is the Boltzmann constant (ergs/degree), and T is the absolute temperature.

A test of the validity of eq. (5) is presented in Figure 1, where the range of application was found to vary from 3.92×10^5 to 9.80×10^5 dynes for

the rupture force. At lower forces, abnormally high rupture times were recorded due to the cold flow of the sample. The measurement was affected by both the cohesive strength and autohesive bonding. Moreover, at higher breaking forces, the experimental points tended to rise above the dotted line, which indicated that chain entanglement was becoming increasingly more important at short rupture times. The time for the surface separation was thus greater than expected from eq. (5).

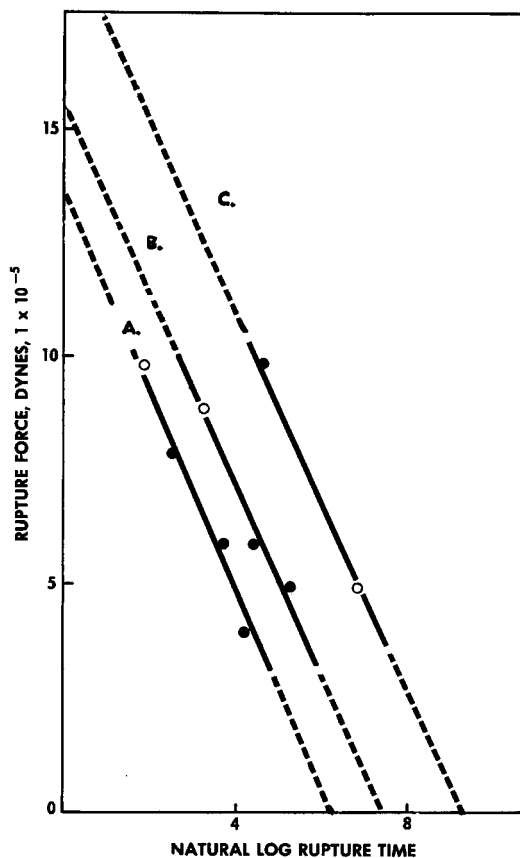


Fig. 1. Tackmeter measurements of raw SBR 162 at 21.5°C. The three curves represent a contact time of 400 sec. and contact force of: (A) 2.94×10^5 dynes, (B) 5.88×10^5 dynes, and (C) 9.80×10^5 dynes for equilibrated samples.

The contact pressure was found to be a significant factor in promoting autohesion as shown by curves A, B, and C in Figure 1. As the contact pressure was increased, the rupture time was also greater at a given rupture force. A linear relationship was then obtained between the contact force and the rupture time for a complete bond at $F_B = 0$ which is illustrated in Figure 2. Particularly interesting is the finding that the slopes of curves A, B, and C in Figure 1 are equal to each other and equal to the negative

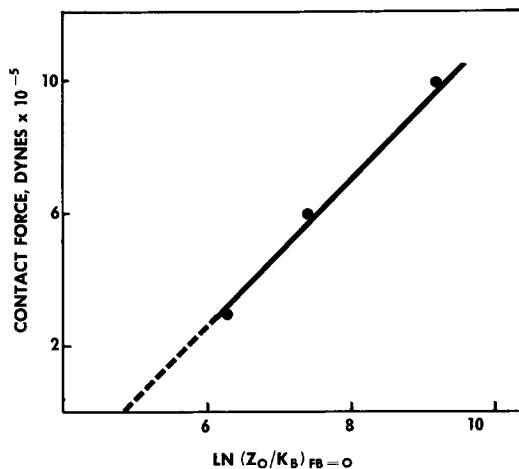


Fig. 2. Intercepts in Figure 1 at $F_B = 0$ plotted against the contact force for equilibrated raw SBR 162 at 21.5°C. The intercept at $F_C = 0$ is the natural log of t_0 , the average lifetime of the autohesive bond.

slope of the line in Figure 2. This means that the processes involved in forming an autohesive bond are the exact reverse to rupturing the bond.

Again referring to Figure 2, one notes that the extrapolated part of the line starts at a contact force of 2.94×10^5 dyne. It was impossible to obtain consistent measurements at lower contact forces F_C , probably owing to poor surface contact. However, the intercept at $F_C = 0$ is real and agrees in value with the evidence to be presented.

It is Figure 2 that provides experimental evidence for eq. (6):

$$F_C = K \ln (Z_0/k_B)_{F_B=0} - K \ln t_0 \quad (6)$$

where F_C is contact force (dynes) and t_0 is the lifetime of an elementary area bond (seconds). The addition of eqs. (5) and (6) gives:

$$F_B - F_C = -K \ln t_B + K \ln t_0 \quad (7)$$

and

$$t_B = t_0 \exp \{ (F_C - F_B)/K \} \quad (8)$$

The test for eq. (7) is presented in Figure 3, where the tackmeter data are fairly well represented up to $F_B - F_C = 6.86 \times 10^5$ dynes. The intercept for curve *C* at $F_B - F_C = 0$ has the same value as the extrapolated portion of the line in Figure 2. It is, therefore, possible to obtain a direct measurement of t_0 from the time clock which registers the time necessary for rupturing a complete bond by having $F_B = F_C$.

Since t_0 is independent of pressure, it provides a useful parameter to measure the time required to form a complete area bond. If one measures t_0 over a large range of contact times and at smaller intervals than shown in Figure 3, one can plot characteristic curves of contact time versus

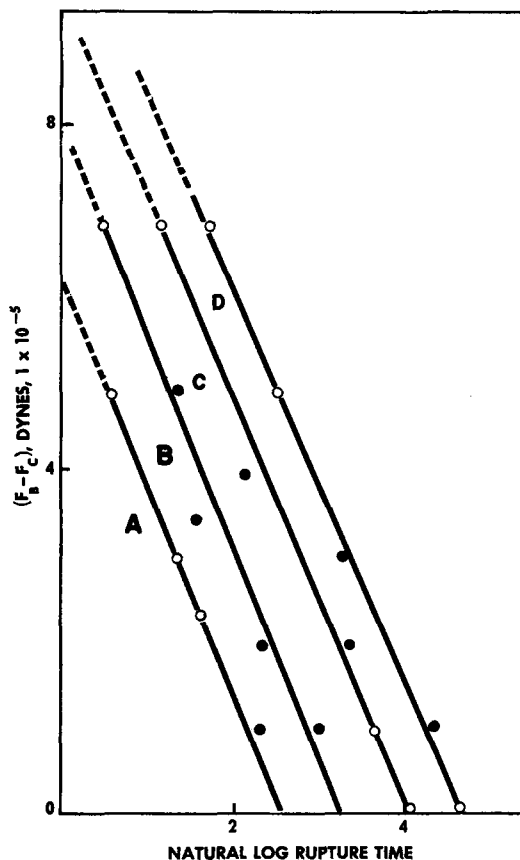


Fig. 3. Application of eq. (7) to tackmeter measurements: (A) contact time 20 sec.; (B) contact time 50 sec.; (C) contact time 100 sec.; (D) contact time 200 sec. Samples equilibrated to a constant force of 2.94×10^6 dyne.

rupture time for the development of the autohesion, as shown in Figures 4-6 for raw SBR 162. The real value of t_0 is represented by the line which is almost vertical to the abscissa. The point of inflection gives the earliest contact time to form the bond. In Figure 3, this is represented by the intercept given by the line D. Intercepts obtained from the lines A, B, and C are the rupture times t_0' of a fractional or partly developed area bond.

The experimental information obtained on the autohesion for raw SBR samples 160, 161, 163, and 164 under equilibrium conditions are shown in Figures 7, 8, 9, and 10, respectively. These graphs show in addition to what has been described, the development of a more complex type of bonding, perhaps a three-dimensional bond, which is represented by an exponential curve. The area bond is seen to have been completed in contact periods as short as 32 sec. for SBR 160 and as long as 600 sec.

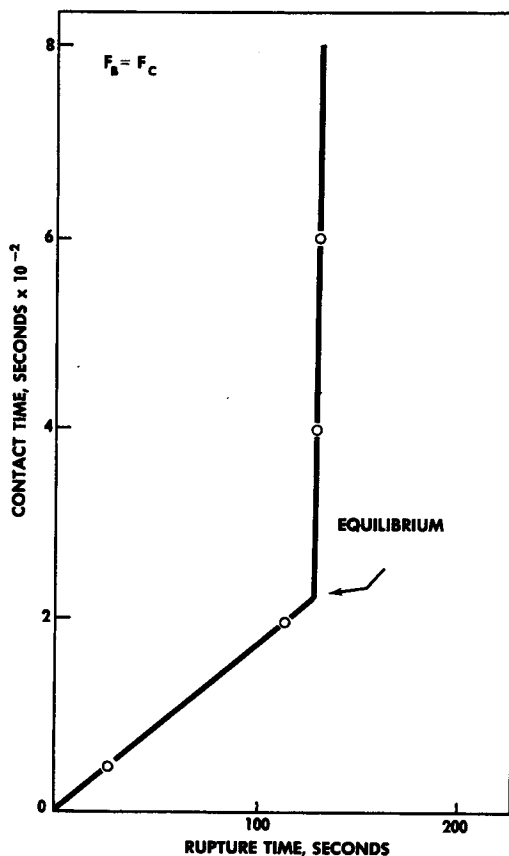


Fig. 4. Development of autohesive area bonding for raw SBR 162 equilibrated to the contact force of 2.94×10^6 dyne at 21.5°C.

for SBR 164. The more complex cohesive bond begins to form in contact periods as short as 150 sec. as in the case of SBR 160.

Equations (1)–(8) do not express any quantitative relation between contact time and the rate of bond development. The equations refer to properties of a completely formed area bond which is undergoing bond rupture.

Non-Equilibrium Conditions

The formation of an autohesive area bond under non-equilibrium conditions involves the usual joining together of rubber surfaces by the application of an instantaneous pressure. The surfaces must bond through the processes of molecular segmental migration and reduction in the molar free (hole) volume. The change in the molar free volume with time distinguishes this method of bonding as non-equilibrium in nature. The derivation of the kinetic equations which expresses the contact time for bond formation

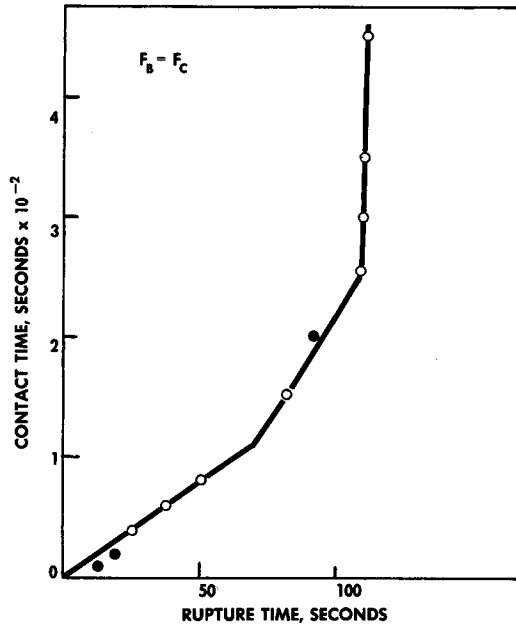


Fig. 5. Development of autohesive area bonding for raw SBR 162 equilibrated to the contact force of 5.88×10^6 dyne at 21.5°C .

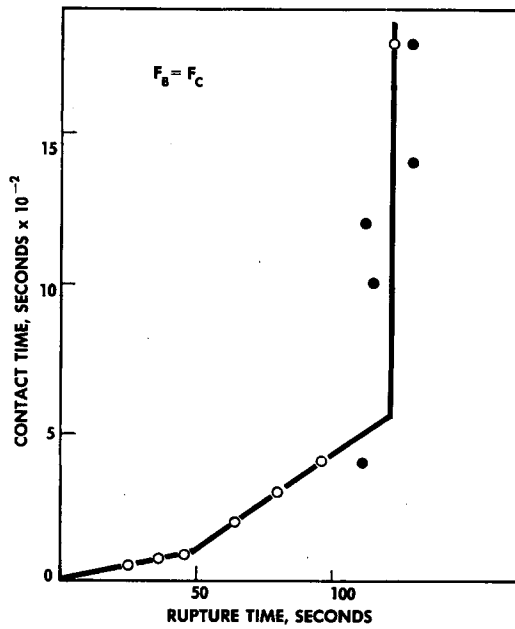


Fig. 6. Development of autohesive area bonding for raw SBR 162 equilibrated to the contact force of 9.80×10^6 dyne at 21.5°C .

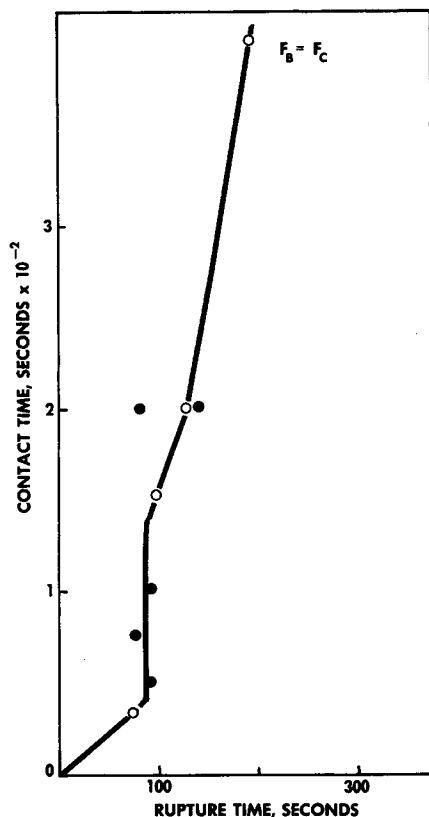


Fig. 7. Development of autohesive area bonding for equilibrated raw SBR 160 at 21.5°C.
 $F_C = F_B = 9.80 \times 10^6$ dyne.

as a function of the bond rupture time is given below, and has been experimentally verified to follow first-order kinetics.

Starting with the general rate equation for the change in area bonding with time expressed by eq. (9):

$$dz/dt = k_A[(N_0)^{2/3} - Z] - k_B Z \tag{9}$$

where k_A and k_B are rate constants for the formation and disappearance of area crosslinks, the concentration of area crosslinks C at a steady-state condition, $dz/dt = 0$, is:

$$\begin{aligned} C &= Z/(N_0)^{2/3} \\ &= k_A/(k_A + k_B) \approx k_A/k_B \\ &\approx t_B \exp \{W_B/kT\}/t_C \exp \{W_C/kT\} \\ &\approx (t_B/t_C) \exp \{(F_B - F_C)/K\} \approx t_0'/t_C \end{aligned} \tag{10}$$

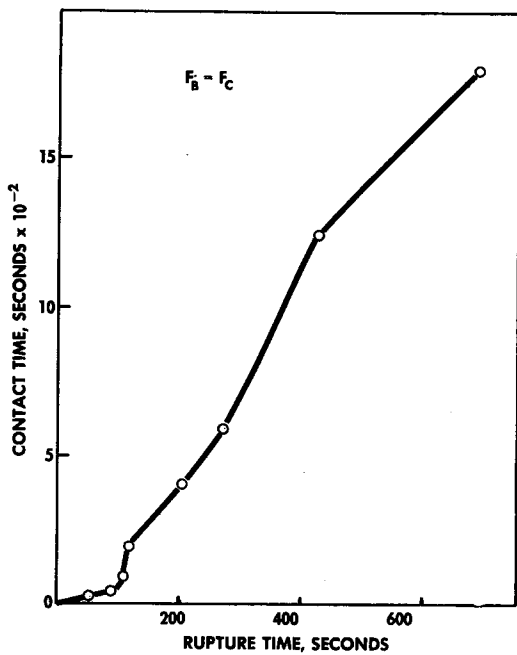


Fig. 8. Development of autohesive area bonding for equilibrated raw SBR 161 at 21.5°C.; $F_C = F_B \geq 12.74 \times 10^6$ dyne.

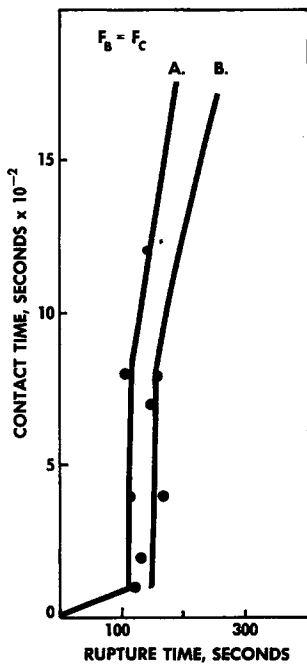


Fig. 9. Development of autohesive area bonding for equilibrated raw SBR 163 at 21.5°C.; $F_C = F_B = 4.90 \times 10^6$ dyne: (A) initial experiments; (B) re-run of same samples.

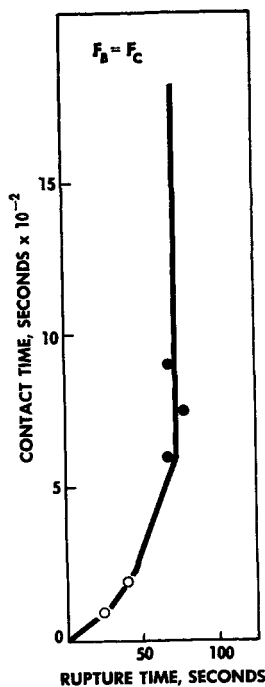


Fig. 10. Development of autohesive area bonding for equilibrated raw SBR 164 at 21.5°C.; $F_C = F_B = 4.90 \times 10^6$ dyne.

where t_0' is the rupture time for an incomplete (or fractional) bond development when $F_B = F_C$ at the contact time t_C . Applying the integrated first-order equation,

$$C = C_0 \exp \{-k_1 t_0'\} \tag{11}$$

gives eq. (12) by substitution:

$$\ln t_C = k_1 t_0' + (\ln t_0' t_C^*/t_0) \tag{12}$$

where t_C^* is the contact time to form a complete area bond having the rupture time t_0 . When $\ln t_C = 0$,

$$\ln t_0' t_C^*/t_0 = k_1 t_0$$

and

$$\ln t_C = k_1 t_0' + k_1 t_0 \tag{13}$$

A more generalized equation would involve the occurrence of stresses as those produced during tackmeter measurements:

$$\ln t_C = k_1 t_B \exp \{(F_B - F_C)/K\} + k_1 t_0 \tag{14}$$

which can be rearranged into another useful form:

$$F_B - F_C = -K \ln t_B + K \ln t_0' \tag{15}$$

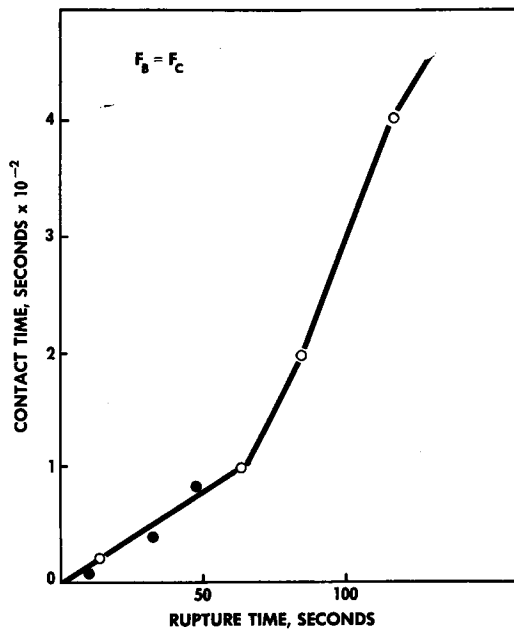


Fig. 11. Development of autohesive area bonding for non-equilibrated raw SBR 162 at 21.5°C. by plot of t_0' against contact time. The contact force is 4.90×10^6 dyne and is equal to the rupture force.

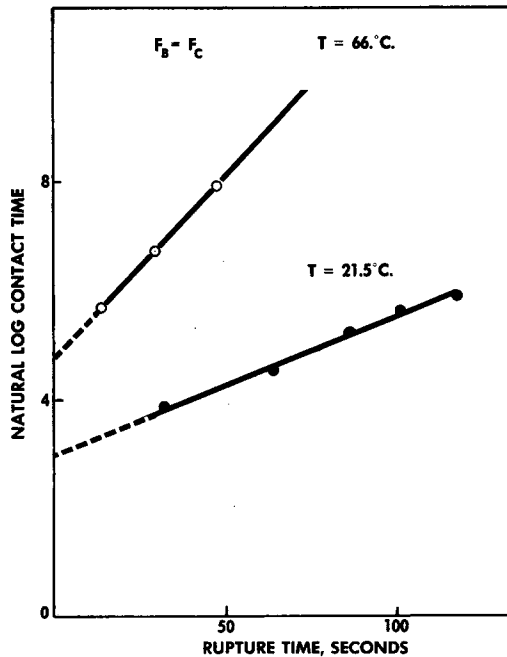


Fig. 12. Linear relationship between the natural log of the contact time versus t_0' for non-equilibrated raw SBR 162 at temperatures of: 66°C. and 21.5°C.; $F_C = F_B = 4.90 \times 10^6$ dyne.

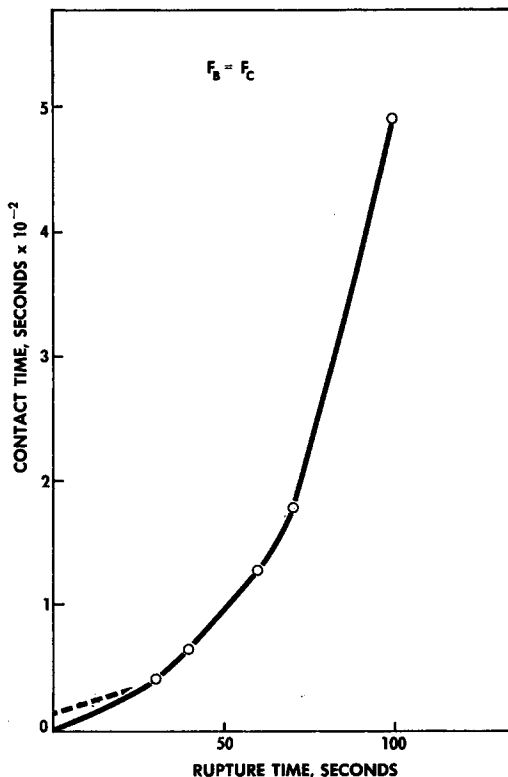


Fig. 13. Development of autohesive area bonding for nonequilibrated raw SBR 160 at 21.5°C.; $F_C = F_B = 14.75 \times 10^6$ dyne.

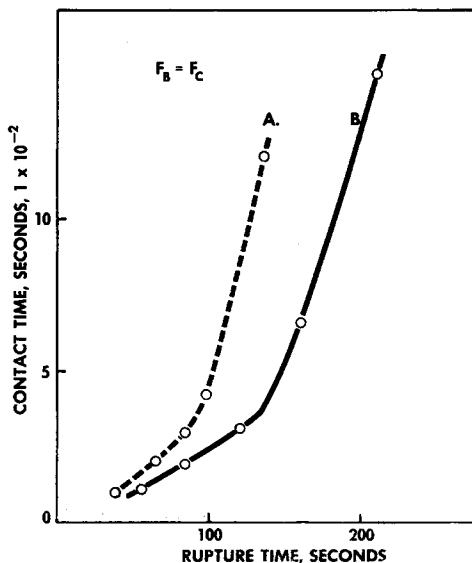


Fig. 14. Development of autohesive area bonding for non-equilibrated raw SBR 161 at 21.5°C.: (A) is correct measurement for $F_C = F_B \cong 12.74 \times 10^6$ dyne; (B) cold flow of sample with $F_C = F_B = 9.80 \times 10^6$ dyne.

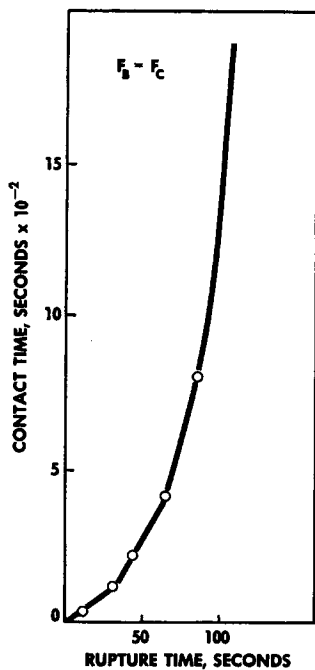


Fig. 15. Development of autohesive area bonding for non-equilibrated raw SBR 163 at 21.5°C.; $F_C = F_B = 9.80 \times 10^6$ dyne.

Equations (13) and (14) are used to determine the contact time required to form a complete bond. Plotting $\ln t_C$ versus t_0' or $t_B \exp \{(F_B - F_C)/K\}$ gives a straight line having slope k_1 and intercept $k_1 t_0$. Consequently the required contact time to form the completed bond is given by:

$$\ln t_C^* = 2k_1 t_0 \quad (16)$$

In order to determine the energy required to break the autohesive area bond the parameters, work needed for bond rupture W and W_{\max} , are defined as given in eqs. (17) and (18).

$$W = f_B a^*/2.022 A_s Z_0 \quad (17)$$

where

$$f_B = K \ln t_0 + F_C$$

and

$$W_{\max} = W - \Delta F^\ddagger \quad (18)$$

where F is the kinetic free energy change,

$$F^\ddagger = -RT \ln (k_B/k_A) = -RT \ln (N_0)^{2/2}/Z_0 \quad (19)$$

The conventional treatment of the tackmeter data involves the calculation of the work required to rupture the autohesive area bond. The

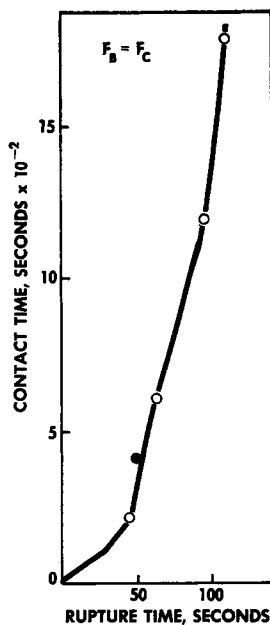


Fig. 16. Development of autohesive area bonding for non-equilibrated raw SBR 164 at 21.5°C.; $F_C = F_B = 7.84 \times 10^6$ dyne.

comparison of autohesive strength between various polymers in a practical situation depends more often, on the contact time restrictions. Consequently, the work done will depend on the degree of bond development. In this case, the work required to break the complete area bond is multiplied by the ratio (t_0'/t_0) , where (t_0'/t_0) represents the fraction of bond area developed at the desired contact time.

A useful relation between the work necessary to rupture the area bond and the rupture time t_0 for an idealized zero stress state can be obtained from eq. (8). Under these conditions, the value for t_B has the intercept value of 1 sec., which gives:

$$t_0 = \exp \{W_0/RT\} \quad (20)$$

This equation can be used to calculate W_0 at the temperature of the experiment and t_0 for the other temperatures, provided the elastomer does not undergo a physical transformation, such as crystallization, freezing, or, conversely, melting.

The experimental data for the raw samples are presented in Figures 11–16 for the nonequilibrium conditions. The curves were of the exponential type and therefore did not show the sharp breaks at the completion of the area bond development which were common for the equilibrated samples.

Logarithmic plots of the experimental nonequilibrium curves are linear as illustrated in Figure 12 for SBR 162 at 21.5 and 66°C. These lines give

TABLE II
Kinetic Analysis of the Autohesive Area Bond for Raw SBR Samples, Either Unstressed or Stressed at Contact Pressure 8.82 psi and Ruptured at a Pressure of 4.41 psi

Parameter	Symbol	Raw SBR at 21.5°C.					Raw SBR	
		SBR 160	SBR 161	SBR 162	SBR 163	SBR 164	162 at	66°C.
Time required to rupture a complete bond under stress, sec.	t_B	159	203	1174	458	187	698	
Average lifetime of a bond at zero stress, sec.	t_0	82.3	111	125	113, 148	74	—	
From equilibrium data		85.5	137	117	108	68.2	73	
Time required to form a complete bond, sec.	t_C^*	298	1213	428	1766	986	16,300	
From non-equilibrium data		32	50	235, 250, 550	100	50,600	—	
Constant from equilibrium and non-equilibrium data, dynes $\times 10^{-6}$	K	7.44	8.10	2.15	4.33	5.28	2.17	
Bonds/cm. ² $\times 10^{-10}$	Z_0	2.92	3.70	0.843	1.70	2.07	0.739	

evidence that the time required to form a complete autohesive area bond is increased from 428 sec. at 21.5°C. to 16,300 sec. at 66°C. The value for t_0 is 117 sec. at 21.5°C. and 73 sec. at 66°C. The dotted lines in Figures 12 and 13 are used to indicate that the intercept value for eq. (13) is an extrapolation of experimental values. The experimental points deviate sharply from the dotted line at low contact times. The experimental data for contact times exceeding 400 sec. for the curve in Figure 12 at 21.5°C. are not shown. These data give a line of lesser slope possibly indicating the transition of the area bond to a more complex structure.

Although the values for t_0 and t_0' are independent of pressure, it is possible to introduce an experimental error due to the cold flow of the sample. In this instance, the values of t_0' will vary with the force used to break apart the surfaces, as shown in Figure 14. As the breaking force decreases in value, the value for t_0' increases for a particular contact time. Consequently, the effect of the cohesive bond strength becomes more dominant. This error is rectified by measuring t_0' with increasing breaking force until it remains constant at a certain contact interval. Measurements are then obtained for t_0' over a wide range of contact times.

DISCUSSION

A characterization of the five SBR samples on the basis of the above equations is presented in Table II. The information shows that there is good agreement between the values for t_0 and K for the equilibrium and non-equilibrium conditions. The differences in the t_0 values obtained from these conditions for each sample indicate that the experimental error in these measurements has an estimated standard deviation of 1.83%. The contact times required to form the elementary area bond are considerably reduced by equilibrating the samples under the contact pressure prior to joining them. Although the contact time needed to form a complete area bond is less for SBR 160, this sample does not offer the best autohesive properties with regards to the energy for bond rupture.

When referring to the change in kinetic free energy, work, and maximum work for bond rupture in Table II, it is SBR 162 that requires the largest energy exchange. Particularly in the stressed state, this sample requires the largest increase in work energy for bond separation. Apparently, the samples having either a higher or lower number-average molecular weight do not show the same improvement under compression. The data also indicate that SBR 162 has not only the largest bond area, but also the largest number of segments per bond. This kind of bond seems to be related in size to a supermolecular unit. In accord with the above energy considerations, the rate constants for bond rupture, k_B and k_1 , are the lowest in value for SBR 162.

It is possible that, as the number-average molecular weight of these raw SBR samples is increased from 0.74×10^5 to 1.33×10^5 , the lattices become less isotropic and more nematic, i.e., they have orientation without

periodicity. This type of phase would provide a micropore volume which would permit easy penetration of the polymer segments. The compression of SBR 162 may cause segmental flow into these micropores with an attendant reduction in void volume. The presence of a micropore system would also permit flowing and closer alignment of a larger number of segments at internal surfaces with higher contact pressures. The rupture of these surface bonds would also require additional energy over the unstressed state conditions.

At molecular weights higher than 1.33×10^5 for these raw SBR samples, it is possible that the penetration of polymer groups into the micropore volume is obstructed by the presence of additional crosslinks and less terminal groups. The autohesion properties then reflect the movement of smaller chains having greater fluidity; consequently a decrease in autohesion strength would result at 21.5°C.

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Résumé

L'analyse cinétique de l'autoadhésion de caoutchoucs bruts, froids et d'émulsion SBR, ayant des poids moléculaires moyens en nombre variant de 74.000 à 169.000 a été étudiée en utilisant le tackomètre modifié de Wallace. L'autoadhésion a été trouvée être un processus réversible suivant une cinétique de premier ordre par rapport à la formation des liens de surface élémentaires. L'application de pressions de contact plus élevées au cours de l'autoadhésion cause une augmentation du travail cinétique, de la force de la liaison et du temps requis à la rupture du lien. Un aspect pratique de l'étude montre que la durée de contact nécessaire à la formation d'un lien de surface complet est réduit par: (1) le traitement des échantillons de façon à faire disparaître les tensions internes; (2) la production de surfaces propres et très égales; (3) des échantillons équilibrés sous pressions avant de les unir; (4) abaissement de la température de l'échantillon. Le SBR brut offrant les propriétés autohésives potentielles les meilleures avait un poids moléculaire moyen en nombre de 133.000 et ML-4 (212°F) de 66.

Zusammenfassung

Eine kinetische Analyse der Autohäasion von rohem Kalt-Emulsions-SBR-Kautschuk mit Zahlenmittel-Molekulargewichten von 74000 bis 169000 wurde mit dem modifizierten Wallace-Tackmeter unternommen. Die Autohäasion erwies sich als reversibler Prozess mit einer Kinetik erster Ordnung in Bezug auf die Bildung der Elementar-Flächenbindungen. Die Anwendung grösserer Kontaktdrucke während der Autohäasion führt zu einer Erhöhung der kinetischen Arbeit, der Bindungsfestigkeit und der zum Bruch

der Bindung erforderlichen Zeit. Als praktischer Aspekt der Untersuchung ergibt sich, dass die zur Bildung einer vollständigen Flächenbindung notwendige Kontaktdauer durch: (1) Temperung der Proben zur Aufhebung innerer Spannungen; (2) Erzeugung reiner und sehr glatter Oberflächen; (3) Einstellung des Druckgleichgewichts der Proben vor ihrer Vereinigung; (4) Erniedrigung der Probentemperatur verringert wird. Roh-SBR mit den besten potentiellen Autohäsioneigenschaften besitzt ein M_n von 133.000 und ML-4 (212°F) von 66.

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