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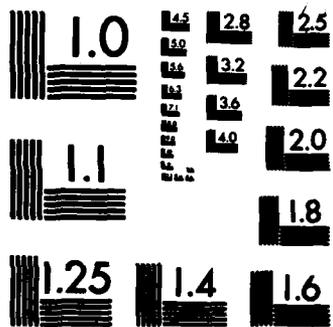
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Annual Technical Report  
THE ROLE OF CHEMICAL BONDING IN ADHESION  
A. N. Gent and P. Dreyfuss

Institute of Polymer Science  
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April, 1983

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Work over the past several years has shown that understanding the effect of chemical bonding on adhesion requires a knowledge of a variety of chemical and physical phenomena. Accordingly, current studies have covered a broad range of related topics. Those investigated during the period from March 1, 1982 to February 28, 1983 are summarized in this report, which describes eleven technical reports, fifteen papers and eleven presentations made during the year.		

Block #19 (cont.)

swelling equilibria, tearing, velocity of retraction, velocity of sound

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## 1. Research Activities

Work during the past several years has shown that understanding the effect of chemical bonding on adhesion requires knowledge of a variety of chemical and physical phenomena. Accordingly our studies have covered a broad range of related topics. Among them are theoretical and experimental studies of the mechanisms of failure of model elastomeric networks and of elastomers containing rigid inclusions. Elastomers of varying chemical structure and degree of crosslinking, and rigid inclusions of varying size, shape, surface treatment and volume concentration, have been used in these studies. Some measurements of the velocity of high-speed cracks in simple elastomeric materials have also been made, and accounted for in terms of the speed of a stress pulse in stretched materials. Efforts to explain an observation that silicone rubber adheres spontaneously to itself, to metal, or to glass, eventually led to the discovery of an unexpected slow decomposition reaction, and a parallel bonding reaction. In addition, a detailed study of the role of amines in the adhesion of polybutadiene to glass and to some metals was made. Results were obtained both with high molecular weight bulk polybutadiene and with liquid polybutadiene as the soft segment of a polyurethane.

Results of recent studies have been described in detail in eleven technical reports issued during the period March 1, 1982 to February 28, 1983. Brief summaries are given in the next section of this report. The 15 publications and 11

presentations related to this work and made during the same period are also listed below.

Experiments are continuing in three main directions. A more detailed examination is being carried out of the processes of failure in an elastomer containing a single rigid inclusion. It is hoped also to extend these studies to multiple inclusions and to shapes other than spherical. A second series of investigations is being conducted on the viscoelastic contributions to the strength of adhesion and, particularly, into the effect of the presence of non-crosslinked species on the adhesion of simple crosslinked elastomers. A third series of investigations involves studies of adhesion in the polybutadiene/polyurethane system. Chemical and physical treatments of the surfaces prior to application of the adhesive layer have been found to have marked effects on the durability of the bond formed. Accordingly, it is intended to continue studies of the durability of different kinds of chemical and physical bonds when exposed to water at various temperatures and in some other environments. Similar studies to those described in this report for the polybutadiene/polyurethane system are also being conducted with polyurethanes containing polyetherglycols such as BAMO as the soft segment.

## 2. Results and Implications

### Studies of Failure Processes in Elastomeric Materials

a) Tear strengths have been measured for a wide variety of elastomeric molecular networks under threshold conditions; i.e., at high temperatures, low rates of tearing, and with swollen samples. For all of the polymers examined, the threshold tear strength was found to be proportional to the square root of the average molecular weight  $M_c$  of network strands, in agreement with theory. However, for the same  $M_c$  and hence for similar values of elastic modulus, different polymers showed major differences in threshold tear strength. The tear strength of polydimethylsiloxane networks was only about one-third as large as that for networks of polybutadiene and cis-polyisoprene and the values obtained for polyphosphazene networks were only about one-fifth as large, at the same  $M_c$ . These striking differences are attributed to differences in network strand length and extensibility for the same molecular weight. The threshold tear strengths are shown to be in satisfactory quantitative agreement with theoretically-predicted values on this basis (15).\*

b) Polydimethylsiloxane (PDMS) networks having strands of molecular weight in the range 11,000-36,000 have been prepared by endlinking linear PDMS molecules of these molecular weights with a tetrafunctional linking agent. Absorption of a series of homologous linear PDMS molecules by the resulting PDMS networks has been investigated. The diffusion coefficients at 70° were

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\* The numbers correspond to the list of technical reports below.

found to be rather large,  $1 \times 10^{-12} - 6 \times 10^{-12} \text{m}^2/\text{s}$ , and approximately inversely proportional to the molecular weight of the diffusing liquid, over the range 5,000-38,000. The amount of liquid absorbed at equilibrium was relatively small, 10% - 80%, in good agreement in all cases with an especially simple version of the Flory-Huggins theory when the heat of mixing is made vanishingly small and only entropic terms are retained (20).

c) Measurements have been made of the velocity of sound and the velocity of free retraction for stretched strips of vulcanized natural rubber. Both of these velocities are found to increase markedly with increasing strain, in agreement with earlier work. The velocity of sound is shown to be related to the appropriate modulus of elasticity, defined by the slope of the curve relating true stress to tensile strain. Values obtained range from about 50 to about 800 m/s. The effects of prior stretching and of stress relaxation on the velocity of sound are shown to arise from corresponding changes in the modulus of elasticity at a given strain. The velocity of free retraction is shown to be directly related to the velocity of sound in the stretched strip, and to the imposed tensile strain increasing from zero up to about 100 m/s at high strains (16).

d) Measurements are reported of the limiting velocity of a running crack in biaxially stretched sheets of unfilled and carbon-black-filled natural rubber. The crack velocity was found to increase with the cleavage strain  $e_y$  and also with

the strain  $e_x$  parallel to the direction of tearing, reaching values of over 100 m/s at the highest strain levels employed. These crack velocities are shown to be close to those predicted by Mott's theory, i.e., about one-third of the velocity of sound, when a strong strain-dependence is recognized for the velocity of sound in rubber (17).

#### Studies of Filled Systems

a) A literature survey of the effect of the particle size of nonreinforcing fillers on the mechanical properties of elastomers and a critical evaluation of the literature results compared to new data from the authors' laboratory were made. The effect of filler content was also considered. The study demonstrated that qualitatively mechanical properties of filled elastomers in general are a complex function of the filler's particle size (surface to volume ratio) and loading (distance between particles). Quantitatively, properties were dependent on the system being studied. Relative modulus, relative breaking elongation, relative breaking stress and swelling ratios were functions of the reciprocal size of the filler up to a particle size of about 140  $\mu\text{m}$ . With particles of larger diameter the influence on these properties was smaller. When the distance between particles is smaller than their size, interactions in the volume surrounding the particles may become the dominant variable (21).

#### Properties of Silicone Rubber

a) Lightly-crosslinked silicone rubber has been found to bond spontaneously to polar substrates and to itself. The strength

of adhesion increases linearly with time, eventually reaching the fracture strength of the rubber. The process is accelerated by raising the temperature (activation energy  $\approx 60$  kJ/g-mole), by the presence of moisture, and by ammonia vapor. It is tentatively attributed to hydrolytic decomposition of the polymer leading to the formation of reactive groups which interlink with surface groups, probably hydroxyls, on polar substrates, or with each other in the case of self-adhesion (18).

b) The sol content of previously-extracted samples of lightly-crosslinked silicone rubber has been found to increase during prolonged storage under relatively mild conditions. Simultaneously, the tensile stress slowly decreases in samples held stretched, and the equilibrium degree of swelling increases somewhat. Thus, the polymer network appears to undergo slow decomposition. This process is accelerated by moisture, by ammonia vapor and by raising the temperature of storage. It is slowed down by prior treatment of the polymer with a silazane reagent which reduces the number of residual OH groups. It is therefore attributed to hydrolytic decomposition of the polydimethylsiloxane molecules, initiated by OH groups (19).

#### Role of Amines in Adhesion of Polybutadiene to Substrates

An attempt to elucidate the role of amines in general, and of 3-aminopropyltriethoxysilane in particular, has been made in several stages.

a) First, the effect was examined of adding small amounts of amine on the physical properties of dicumylperoxide-cured bulk polybutadiene. The molar amount of amine added was of the same

order of magnitude as the amount of peroxide used. When aromatic or cyclic amines were added, maximum stress at break and minimum swelling ratios were observed when the number of alkoxide radicals generated was equal to the number of hydrogen atoms on the nitrogen of the amines. The order of reactivity was thus no amine < aniline < piperazine < m-phenylenediamine. The greatest effect of aliphatic amines also occurred when the concentration of primary amino groups was equal to the concentration of peroxide; but the behavior was complex and dependent on the backing against which samples were molded. The higher rigidity and reinforcement of the polybutadiene in the presence of low concentrations of amines was attributed to formation of chemical bonds between the amines and the polybutadiene (22).

b) Secondly, the reactions that can occur between amines and glass were investigated. Near infrared, infrared and nuclear magnetic resonance spectroscopy studies were carried out of the interaction of amines with silanol groups, usually under ambient conditions but sometimes when heated in air. Additional evidence was obtained from gas liquid chromatography, mass spectrometry and elemental analyses. In order to assure a sufficiently high concentration of silanol groups, triethylsilanol and fumed silica were used as models for the glass surface. The mechanism of interaction of aliphatic amines and of the aminosilane was different from that of aromatic amines. Most notably, reaction of aliphatic amines with carbon dioxide in the air and/or dissolved/adsorbed in/on the silanol occurred almost instantaneously, whereas the corresponding reaction of the aromatic amines with

carbon dioxide was not observed under the experimental conditions used. The carbamates formed underwent further reactions much more rapidly than either the simple aromatic amines or the unmodified aliphatic amines (23).

c) Next, the reactions that occur between the amines and the polybutadiene were studied, using near infrared to examine the influence of amines on the reactions of model olefins with dicumyl peroxide. The model olefins were chosen to be representative of typical internal and external double bonds found in polybutadiene. The study has shown that in addition to serving as radical traps the amines participate in other ways. Exactly what occurs depends on the structure of the amine (24).

d) The effect was also studied of amine structure and concentration on the strength of adhesion of peroxide cured polybutadiene to a glass surface. Adhesion of polybutadiene to glass surfaces is enhanced by incorporation of small amounts of amines into the elastomer. An 80-fold increase in the work of adhesion was observed in the presence of 3-aminopropyltrimethoxysilane. These results are interpreted in terms of information given in previous reports (25).

e) Amines in general and 3-aminopropyltriethoxysilane in particular have been shown to improve the adhesion of a polyurethane to glass. At equivalent concentrations of amine in the solutions used to treat the glass the order of increase in the work of adhesion was 3-aminopropyltriethoxysilane >> 1,4-diaminobutane o p-phenylenediamine > piperazine >> aniline. The polyurethane was prepared from ARCO's hydroxyterminated polybutadiene, R-45HT,

toluene diisocyanate, trimethylolpropane and N,N-bis(2-hydroxypropyl)aniline. The ratio ( $[-\text{NCO}]/[\text{total OH}]$ ) was kept constant and equal to 1.0. The swelling ratio, after curing, also remained constant. As the -NCO content compared to polymer-OH increased, the adhesion of the polyurethane to glass increased initially, passed through a maximum for prepolymers with 6% excess NCO, and then decreased again. Elongation at break behaved similarly but ultimate tensile strength reached a maximum and then stayed constant. The significance of these results is discussed. Adhesion of the polyurethane to chrome-plated steel did not show similar effects (Publication No. 13 in the attached list).

The principal conclusion from the five studies described in this section was that amines become chemically bonded to both the adhesive and the adherend. They contribute positively to the strength of adhesion in direct proportion to the number of reactive groups on the amine provided that it has at least two functional groups and is able to become part of the elastomeric network.

### 3. Technological Significance of the Research

The results described in the preceding part of this report have clear technological implications in several fields. It is now possible to predict from the chemical structure of a proposed new elastomeric polymer what its strength will be under extreme conditions. This is a notable scientific advance.

The maximum speed of movement of rubber sheets and the speed at which they tear has been determined and explained. This

is significant for the design of engineering devices: springs, membranes and seals.

The observed reactivity and spontaneous bonding of supposedly-inert silicone rubber compounds has wide-ranging implications wherever silicone materials are used: in medical applications, as sealants and gaskets, etc.

Small amounts of amines have pronounced effects on the free-radical crosslinking of polybutadiene and on its adhesion to glass. This is relevant to the compounding of elastomers for adhesion and to the reactions of amine-containing coupling agents, widely used for bonding polymers to glass and other substrates.

#### 4. ONR Technical Reports

Report No. 15, A. N. Gent and R. H. Tobias, April, 1982, "Threshold Tear Strength of Elastomers"

Report No. 16, A. N. Gent and P. Marteny, May, 1982, "The Effect of Strain Upon the Velocity of Sound and The Velocity of Free Retraction for Natural Rubber"

Report No. 17, A. N. Gent and P. Marteny, May, 1982, "Crack Velocities in Natural Rubber"

Report No. 18, A. N. Gent and P. Vondracek, September, 1982, "Spontaneous Adhesion of Silicone Rubber"

Report No. 19, P. Vondracek and A. N. Gent, September, 1982, "Slow Decomposition of Silicone Rubber"

Report No. 20, A. N. Gent and R. H. Tobias, October, 1982, "Diffusion and Equilibrium Swelling of Macromolecular Networks by Their Linear Homologs"

Report No. 21, P. Dreyfuss and Y. Eckstein, October, 1982, "Effect of Size of Nonreinforcing Fillers on Mechanical Properties of Elastomers"

Report No. 22, P. Dreyfuss and Y. Eckstein, October, 1982, "Role of Amines in Adhesion of Polybutadiene to Glass Substrates I. Reactions of Amines During Peroxide Curing of Polybutadiene"

Report No. 23, P. Dreyfuss and Y. Eckstein, October, 1982,  
"Role of Amines in Adhesion of Polybutadiene to Glass Substrates.  
II. Reactions of Amines with Triethylsilanol and/or Fumed Silica"

Report No. 24, P. Dreyfuss and Y. Eckstein, October, 1983,  
"Role of Amines in Adhesion of Polybutadiene to Glass Substrates.  
III. Effect of Amines on the Reaction of Peroxide with Olefinic  
Groups"

Report No. 25, P. Dreyfuss and Y. Eckstein, October, 1983,  
"Role of Amines in Adhesion of Polybutadiene to Glass Substrates.  
IV. The Effect of Amine Structure and Concentration on the  
Strength of Adhesion"

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8. P. Dreyfuss and M. P. Dreyfuss, Kirk-Othmer Encyclopedia of Chemical Technology, M. Grayson, Ed., J. Wiley and Sons, New York, 18, 645-670 (1982). "Polyethers, Tetrahydrofuran and Oxetane Polymers"
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5. Lists of Presentations

A. N. Gent, "Aspects of the Adhesion and Fracture of Polymers", presented at:

High Polymer Physics Division, American Physical Society, Dallas, Texas, March 9, 1982

Inaugural Meeting, Tire Society, University of Akron, March 25, 1982

Virginia Polytechnic Institute and State University, Blacksburg, Virginia, April 2, 1982

Akron Polymer Conference on Fracture of Composites, May 20, 1982

Canadian Institute of Chemistry, Toronto, Canada, June 1, 1982

Adhesion Conference, Kent State University, Kent, Ohio, June 10, 1982

IUPAC Symposium on Macromolecules, Amherst, Massachusetts, July 13, 1982

McGill University, Montreal, Canada, September 24, 1982

Adhesion Conference, State University of New York at New Paltz, New York, October 15, 1982

N.S.F. Workshop on Fracture and Damage, Stone Mountain, Georgia, November 23, 1982

P. Dreyfuss:

"Ring Opening Polymerization: Make the Initiator Work for You", presented at the Las Vegas Meeting of the American Chemical Society, March, 1982

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