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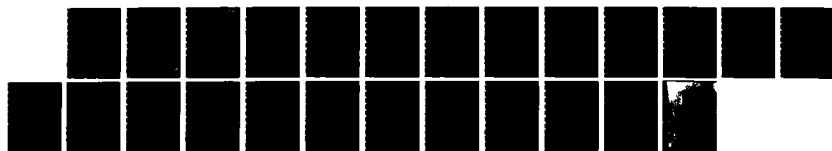
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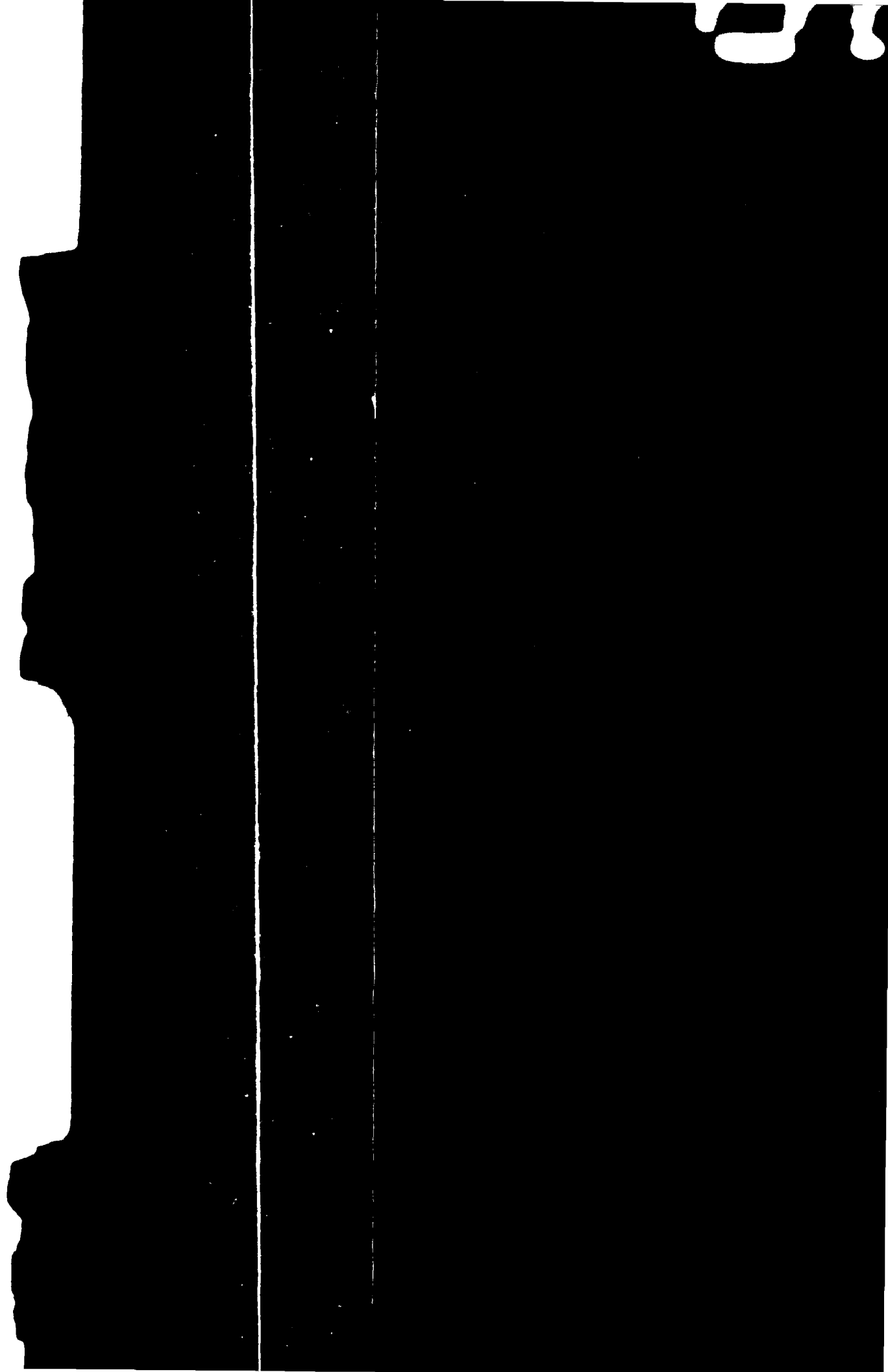
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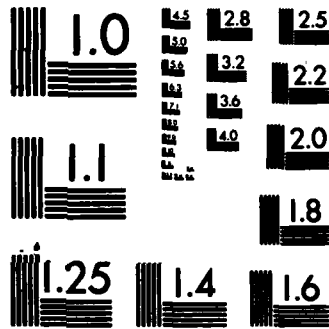
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Annual Technical Report

THE ROLE OF CHEMICAL BONDING IN ADHESION

A. N. Gent and P. Dreyfuss

Institute of Polymer Science
The University of Akron
Akron, Ohio 44325

April, 1984

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Work during the period from March 1, 1983 to February 29, 1984 has been focussed in two main categories, namely, studies of basic failure processes in elastomeric materials and studies of factors affecting the adhesion of various materials to each other. These studies are summarized in this report which describes eight technical reports, 6 publications, and 4 technical presentations made during the year.		

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1. Research Activities

This report covers work carried out during the period from March 1, 1983 to February 29, 1984. Results of studies during this period have already been described in detail in eight technical reports. Only brief summaries are therefore given in this report. The six publications that have appeared during this period and the related presentations are listed at the end of the report.

Projects during this period have been focussed in two main categories; namely basic studies of failure processes in elastomeric materials and studies of factors affecting adhesion of various materials to each other.

The studies of failure processes have dealt with the mechanisms of crack propagation in elastomeric and glassy polymers and of debonding in simple composites. Some factors common to both filled and unfilled systems were found. The results suggested that crazing in glassy polymers and secondary cracking around stress-raising features in elastomers contribute to high toughness in both systems. The conclusion is based on an estimation by the double torsion method of fracture toughness of a crosslinked polybutadiene and a study by electromicroscopy of torn surfaces and of tips of propagating tears in elastomers. Measurements of the tear strength of elastomeric materials under threshold conditions revealed that the enhancement of tear strength due to polysulfidic crosslinks compared to monosulfide or C-C crosslinks was similar to that produced by a reinforcing filler like carbon

black. A substantial contribution from labile crosslinks or bonds was inferred.

Studies of adhesion were carried out in two dissimilar systems. A study of polydimethylsiloxane (PDMS) networks formed by endlinking linear PDMS molecules layer by layer had the goal of estimating the magnitude of the contribution of interfacial entanglements (molecular loops) to the strength of elastomers and the work of detachment of layers. The main conclusion was that permanently "entangled" macromolecular loops appear to have about the same strength as covalently-bonded macromolecules of the same length, and contribute about as much to the threshold strength of the material.

Studies of the adhesion of polyurethanes to varied surfaces have confirmed conclusions reached in earlier studies with bulk polybutadiene and have led to some new ones. As before, it was observed that the degree of enhancement of adhesion could be related to the number of covalent bonds that form between an adhesive and an adherend. A new discovery was that the presence of ionic interfacial bonds is detrimental to the durability of the resulting adhesive bonds in water. Strong bonds reformed if the water was removed. The ionic bonds resulted from the use of N,N-bis(2-hydroxypropyl)aniline, a chain extender possibly used in preparing propellant binders from polybutadiene glycols because of its contribution to good mechanical properties. Good durability could be obtained if the surface was first chemically abraded and then treated with a suitable coupling agent such as n-propylamino-triethoxysilane before the polyurethane was applied.

2. Results and Implications

Studies of Failure Processes in Elastomeric and Glassy Polymers

a) A study of torn surfaces and of the tips of propagating tears in elastomers has been carried out using scanning electron microscopy. Vertical steps separating smooth featureless torn regions are characteristic features of the torn surfaces. They are found to be more frequent and larger in stronger elastomers. They are attributed to the intersection of secondary cracks at the tear tip, displaced somewhat from the general tear plane, and nucleated by inherent stress-raisers. The effective diameter of the tear tip is thereby increased. Stress-raising features are inferred to be present in elastomeric materials at a typical spacing of 10-100 μm . In carbon-black-filled elastomers, the carbon particles themselves are found to nucleate secondary cracks profusely.

b) 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 were similar in all cases, lying in the range 50 - 200 J/m^2 . They appeared to be approximately proportional to $E^{-1/2}$, 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.

c) An estimate of fracture toughness of crosslinked polybutadiene rubber at -180°C has been made using the double torsion method. By using suitable specimen dimensions and strain rates, controlled crack propagation can be achieved, together with a constant compliance to crack length ratio. Strain energy release rates for this polymer, crosslinked with either dicumyl peroxide or sulphur, were found to be an order of magnitude higher than for linear, glassy thermoplastics. Crazeing is considered to contribute to the high toughness observed.

c) A systematic experimental study has been carried out of two distinct failure phenomena, cavitation and debonding, in an elastomer containing a rigid spherical inclusion. Several elastomers were employed containing glass beads of various diameters, ranging from $60\text{ }\mu\text{m}$ to $5000\text{ }\mu\text{m}$, and with chemically-different surfaces. The critical stress for cavitation was found to depend upon Young's modulus E of the elastomer and upon the diameter of the bead. By extrapolation, it was found that the stress for cavitation near an infinitely-large bead is given by $5E/12$, as

predicted by theory. In contrast, the critical stress for debonding decreased somewhat with increasing Young's modulus of the elastomer. This is attributed to a concomitant decrease in the strength of adhesion between the elastomer and the bead surface, due to rheological effects. The stresses for both cavitation and for debonding were found to vary approximately with the negative half-power of the bead diameter. This suggests that a similar Griffith mechanism governs both failure processes when the bead size is small.

A study of cavitation and debonding in the presence of two glass beads was also carried out. As predicted from theoretical considerations, both stresses were found to decrease as the distance between the two beads was decreased, irrespective of the diameter of the bead and Young's modulus of the elastomer. At higher strains, however, a second cavitation process was found to take place at a point midway between the beads. Tensile fracture of the specimen resulted from the unrestrained lateral growth of the second cavity.

e) Griffith introduced a general fracture criterion in 1920: a crack will grow only when enough energy is available in the system to cause further fracture. This simple concept has been applied to various tear processes in elastomeric materials by Rivlin and Thomas and to a variety of adhesive failures by Kendall. Their results are reviewed, with particular reference to the fracture and debonding of elastomeric composites. Two further cases are

then considered: the detachment of an elastic matrix from a rigid spherical inclusion and the pull-out of inextensible cords from an elastic block in which they are embedded.

Adhesion Studies

a) Two studies of the adhesion of a polybutadiene-polyurethane to various substrates have been completed. In the first it was shown that amines in general and 3-aminopropyltriethoxysilane in particular improve the adhesion of a polyurethane to glass. (Glass was used as a model for a propellant surface such as ammonium perchlorate. Even though glass is not an ideal model for a crystalline material, similar results have been obtained and similar conclusions have been reached, when comparable studies have been carried out by ourselves and others, notably H. L. Williams and his coworkers and R. Dick, G. Marvel and R. Mankowski-Favelier.) The polyurethane was prepared from ARCO's hydroxyterminated polybutadiene, R-45HT, toluene diisocyanate, trimethylolpropane and N,N-bis(2-hydroxypropyl)aniline. The ratio ($[-NCO]/[total\ OH]$) 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. At equivalent concentrations of amines in solutions used to treat the glass surfaces the order of increase in the work of adhesion was 3-aminopropyltriethoxysilane $\gg$ 1,4-diaminobutane $\sim$ p-phenylenediamine $>$ piperazine $\gg$ aniline $\sim$ 0. (The chemical reactions

that occur when an amine reacts with an isocyanate in the course of the preparation of a polyurethane are well documented. Interactions of amines with surface-OH groups have been observed also.) As in earlier studies the order of increase in the work of adhesion could be correlated with the number of covalent bonds that can form between the surface and the polyurethane. The level of adhesion was unexpectedly high in all cases. This was attributed to the presence of tertiary amine groups derived from the substituted aniline. These amine groups can react with the surface silanol groups to form ammonium type ionic bonds. Adhesion of the polyurethane to chrome-plated steel was high and not so sensitive to the -NCO content.

In the second study the durability of polybutadiene-polyurethane joints to glass or metals (chrome-steel and aluminum) in water at room temperature was studied. The effects of silane pretreatment of the surface and of surface morphology on adhesion were examined. The presence of interfacial ionic bonds resulted in poor durability of the adhesive bonds in water, although bond strength could be recovered by vigorous drying. A combination of mechanical interlocking to "rough" (chemically abraded) surfaces and covalent bonding to the surface gave much more durable bonds. The durability of the joint was related to the number of chemical bonds that could form between the polyurethane and the surface.

Similar work is in progress with BAMO/THF 5-2. A similar recipe without the N,N-bis(2-hydroxypropyl)aniline is being used.

Initial experiments suggest that adhesion of the resulting polyurethane to various substrates is higher than that of the polyurethane prepared from R-45HT in the same recipe.

b) Polydimethylsiloxane (PDMS) networks have been formed by endlinking linear PDMS molecules. When a second layer is cast on top of a fully-gelled lower layer, the new molecules diffuse into the surface of the lower layer and form molecular loops ("entanglements") in the course of endlinking with themselves. The two layers are then joined only by these macromolecular loops. Measurements have been made of the work required to separate such layers under threshold conditions, i.e., at low rates, high temperatures, and, in some cases, in the swollen state. Values of the work of detachment have been found to be $15\text{--}25 \text{ J/m}^2$, generally about one-half of the work of fracture of the layers themselves, and consistent with the inferred density of interlinking molecular loops at the interface. The values were higher for higher densities, roughly in proportion, and for interlinking molecular strands of higher molecular weight, in accordance with the theory of Lake and Thomas. In the absence of interlinking the work of detachment was extremely small, only about 70 mJ/m^2 .

3. Technological Significance of the Research

The results described in the preceding part of this report have clear technological implications.

The recognition that labile crosslinks or bonds contribute positively toward the strength of elastomers under severe (threshold) conditions makes it possible in principle to design new systems with improved toughness. This is especially significant for the power program, where physical properties such as the toughness of new binders, and propellants, made from them often need improvement.

Failure processes near or at the surface of a rigid inclusion embedded within an elastomeric material are clearly of direct relevance to filled or reinforced elastomers. The present observations suggest that the size of the inclusions is of paramount importance. When they are sufficiently small, then failure is unlikely to occur. However, another failure process takes place when two inclusions are in close proximity. Then, cavitation occurs midway between them when the applied stress exceeds a critical value. This appears to be the major cause of fracture in elastomers containing low concentrations of large-particle fillers.

The adhesion studies are technologically important because they have revealed poor durability in water of adhesive bonds to polyurethanes synthesized using N,N-bis(2-hydroxypropyl)aniline, a chain extender that might be used in the preparation of propellant binders from polybutadiene glycol. The aniline is desirable

since it leads to polyurethanes with substantially inferior mechanical properties. The poor durability results from the lack of interfacial ionic bonds. The work has further demonstrated that durability in water can be significantly improved by the preparation of the surfaces by a combination of chemical and surface treatment with coupling agents. The further observation that the work of detachment of layers of polymer is only by molecular intertwinning (entanglements) has about the same magnitude as the work required to separate covalently bonded layers confirms the importance of physical entanglements in the properties of elastomers and their bonds. It has broad application. If an adhesive is prepared by in situ polymerization, or other procedures are used to manufacture composite structures,

4. ONR Technical Reports

Report No. 26, R. P. Burford, April, 1983, "Fracture Toughness of Polybutadiene at Cryogenic Temperatures"

Report No. 27, A. K. Bhowmick, A. N. Gent and C. T. R. Pulford, April, 1983, "Tear Strength of Elastomers under Threshold Conditions"

Report No. 28, A. N. Gent, April, 1983, "Fracture Mechanics Applied to Elastomeric Composites"

Report No. 29, A. N. Gent and Byoungkyen Park, September, 1983, "Failure Processes in Elastomers At or Near a Rigid Spherical Inclusion"

*Report No. 29A, F. Liang and P. Dreyfuss, December, 1983, "Effect of Amine Surface Treatment on the Adhesion of a Polyurethane to the Surface"

Report No. 30, A. N. Gent and R. H. Tobias, February, 1984, "Effect of Interfacial Bonding on the Strength of Adhesion of Elastomers III. Interlinking by Molecular Entanglements"

Report No. 31, F. Liang and P. Dreyfuss, February, 1984, "Durability of Adhesive Bonds Between Glass or Metal Substrates and a Polybutadiene-Polyurethane"

Report No. 32, A. N. Gent and C. T. R. Pulford, February, 1984, "Micromechanics of Fracture in Elastomers"

Publications

1. Y. Eckstein and P. Dreyfuss, J. Adhesion, 15, 163-178 (1983), "Role of Amines in Adhesion of Polybutadiene to Glass Substrates. II. Reactions of Amines with Trimethoxysilanol and/or Fumed Silica"
2. Y. Eckstein and P. Dreyfuss, J. Adhesion, 15, 179-192 (1983), "Role of Amines in Adhesion of Polybutadiene to Glass Substrates. III. Effect of Amines on the Reaction of Peroxide with Olefinic Groups"
3. Y. Eckstein and P. Dreyfuss, J. Adhesion, 15, 193-202 (1983), "Role of Amines in Adhesion of Polybutadiene to Glass Substrates. IV. The Effect of Amine Structure and Concentration on the Strength of Adhesion"
4. R. P. Burford, J. Materials Science, 18, 3756-3764 (1983), "Fracture Toughness of Polybutadiene at Cryogenic Temperatures"

*Report re-numbered to eliminate duplication of earlier report number.

5. A. K. Bhowmick, A. N. Gent and C. T. R. Pulford, *Rubb. Chem. and Technol.* 56, 226-232 (1983), "Tear Strength of Elastomers under Threshold Conditions"
6. A. N. Gent, *Rubb. Chem. and Technol.*, 56, 1011-1018 (1983), "Fracture Mechanics Applied to Elastomeric Composites"

5. List of Presentations

A. N. Gent:

"Fracture Mechanics Applied to Elastomeric Composites", presented at: (i) ACS Rubber Division Meeting, Toronto, Canada, May, 1983.

(ii) University of Montreal, Department of Mechanical Engineering, October 1983.

(iii) Gordon Research Conference on Composites, Santa Barbara, California, January 1984.

P. Dreyfuss:

"Durability of Adhesive Bonds Between Glass or Metal Substrates and a Polybutadiene-Polyurethane," presented at 1984 JANNAF Propulsion Meeting, New Orleans, LA, February, 1984.

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