

Improved Adhesive Properties and Bonding Performance of HTPB-Based Polyurethane Elastomer by Using Aziridine-Type Bond Promoter

B. O. GERCEL,¹ D. O. ÜNER,² F. PEKEL,³ S. ÖZKAR²

¹ TÜBITAK-SAGE, PK 16 Mamak, 06261 Ankara, Turkey

² Department of Chemical Engineering, Middle East Technical University, 06531 Ankara, Turkey

³ Department of Chemistry, Middle East Technical University, 06531 Ankara, Turkey

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ABSTRACT: The effect of two aziridine-type bond promoters on adhesive and mechanical properties of a hydroxyl terminated polybutadiene-based elastomeric liner used in solid propellant rockets was investigated by varying the concentration to determine the optimum value. The performance of butyleneiminetrimethylaziridine (BITA) was compared with that of tris[1-2-methylaziridinyl]phosphine oxide (MAPO) in the elastomeric liner of otherwise the same composition. The adhesive performance of the elastomer to the composite was determined by using metal-elastomer-composite tensile and peel tests. The adhesive performance of the elastomer to the metal was also determined, this time by using peel and shear tests. The mechanical characterization of the elastomer was done by tensile and hardness tests. A significant enhancement in the bonding performance of the elastomeric liner toward composite propellant and metal case was achieved by optimizing the concentration of bond promoter in the elastomeric composition. All the elastomer compositions with bond promoters BITA and MAPO loadings of 1.0, 1.5, and 2.0 wt % were found to be sufficient for the rocket motor operations because the interfacial adhesive strength of these compositions is higher than the cohesive strength of the composite. Compositions with bond promoter quantities of 1.0, 1.5, 2.0, and 2.5 wt % have better strength values than the others. Liner compositions with the bond promoter BITA give better bonding performance between the composite-metal system and better mechanical properties when compared with the elastomers with the bond promoter MAPO. The best results are obtained in terms of bonding performance and adhesive properties by using the bond promoter BITA in optimized quantities of 1.0 and 1.5 wt % loadings in the elastomer compositions. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 80: 806–814, 2001

Key words: composite; propellant; mechanical properties; curing; HTPB; triol; diisocyanate; bond promoter; aziridine

Correspondence to: S. Özkar (sozkar@metu.edu.tr).

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INTRODUCTION

One of the important structural elements in the case bonded solid propellant rocket motor is the elastomeric liner, which holds the composite fuel in place within the rocket motor case during storage and handling. Furthermore, the presence of an elastomeric liner inhibits propellant deterioration under stresses during operation. The adhesive and mechanical characteristics of the elastomeric liner is an important design parameter in rocket motor applications, because the failure usually occurs at the propellant–liner or liner–motor case interfaces in case bonded systems. Adhesive properties at the rocket motor interfaces can be improved by changing the crosslinking structure of the liner¹ and by using different kinds of bond promoters in the liner compositions.^{2–4} Compounds containing aziridine groups are generally used in propellant composites to enhance the adhesive force in the matrix system, and thus to improve the mechanical and physical properties of the composite.^{5–7}

Experimental studies have shown that bonding agents act by forming a tear-resistant layer around the filler particle, thus preventing peel.⁸ Among those, aziridine-containing compounds were shown to be converted into a polymeric form during the cure process, thus they enhanced the adhesive forces because of the primary chemical bonds of the functional groups to filler and the secondary ion-dipole attraction or hydrogen bonding.⁹ Therefore, the role of the bond promoter is expected to be dependent on the structural features of the aziridine molecule. Despite the widespread use of aziridine-based bonding agents in the composite and aziridine-based bond promoters in the elastomer, the question of how the bonding performance of an elastomeric liner was altered with the aziridine compounds in rocket motors has not been addressed. Herein we report the results of a comparative study on the effect of two different aziridine molecules on the adhesive properties and bonding performance of an elastomeric polyurethane liner. Two aziridine-type bond promoters, tris-[1-(2-methyl)aziridinyl]phosphine oxide (MAPO) and butyleneiminetrimesoylaziridine (BITA), were used in the hydroxyl terminated polybutadiene (HTPB)-based polyurethane elastomer in various percentages. Although MAPO has been commonly used as bond promoter in both the HTPB composite propellants and elastomeric liner systems, there exists no report on the use of BITA in HTPB-based elastomeric lin-

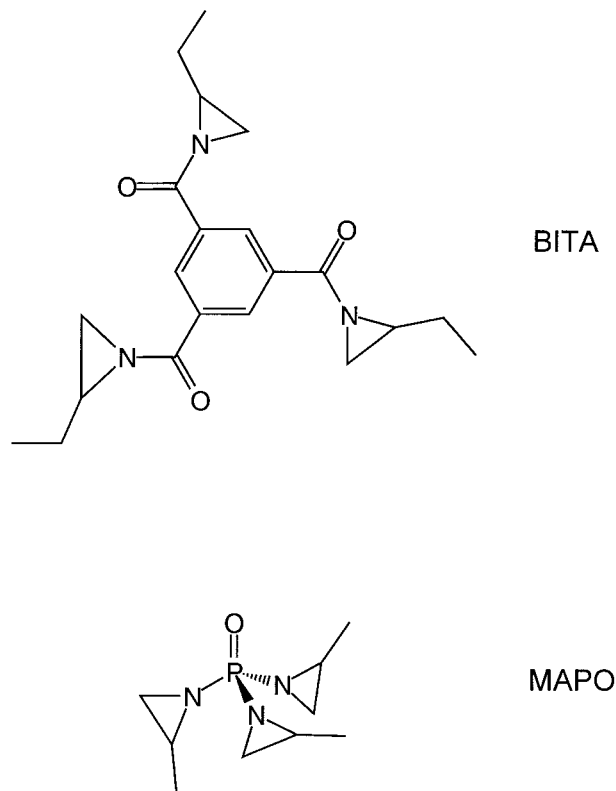


Figure 1 Chemical structures of the bond promoters butyleneiminetrimesoyl-1-(2-ethyl)aziridine (BITA) and trimethylaziridinylphosphine oxide (MAPO).

ers. These two compounds, MAPO and BITA, are compositionally similar, having the same number of aziridine groups which are functionally vital in bond promoting; however, they differ in their spatial arrangement as shown by their molecular structures in Figure 1. MAPO has three aziridine moieties arranged in a pyramidal shape around the phosphorus atom, whereas BITA has a planar arrangement of three aziridine groups. This structural difference can naturally affect their three-dimensional networking ability. Spatially, BITA is expected to be more effective than MAPO which has all of the three aziridine groups oriented facially in one direction, i.e., its reactivity is spatially restricted. With this expectation, a series of HTPB-based elastomeric liners of otherwise the same composition were prepared by using either MAPO or BITA as bond promoter in various concentrations up to 4.0 wt % for a comparative investigation. The adhesive properties and bonding performance of the samples at the interfaces of the metal case-elastomer-composite systems were tested under tensile and peel forces, and the bonds at the interface of the binary elas-

toomer-metal case were examined under peel and shear forces.

EXPERIMENTAL

Materials

HTPB (R-45M, M_n 2700 g/mol, functionality 0.93, antioxidant additive 1 wt %; ARCO Chemical Co., Philadelphia, PA). Isophorone diisocyanate (IPDI) (FLUKA A. G., Buchs, Switzerland), MAPO (ARSYNCO Corp., Carlstadt, NJ), BITA (HX-868; 3M, Cottage Grove, MN), triethanol amine (MERCK, Darmstadt, Germany), carbon black (PRINTEX-U, 25-nm particle diameter, 110 m²/g surface area, 98 wt % carbon content; 1011 Military Basic Restoration Factory, Ankara, Turkey), dioctyl adipate (KIMTAS, Istanbul, Turkey), antioksidant (Irganox 1010; Chemische Fabric Freising, Germany), ferric acetyl acetonate (FLUKA A. G.), triphenylmethane-4,4',4''-triisocyanate (Desmodur-RE; Bayer, Leverkusen, Germany), crystalline ammonium perchlorate (AP) (200- μ m particle size, 99.3 wt % NH₄ClO₄ content; SNPE, Paris, France).

Elastomer Matrix and Composite Matrix Preparation

Before mixing, prepolymer, plasticizer, and reinforcement filler carbon black were conditioned at 65°C for about 16 h to reduce the oxidation sensitivity of the reactants. The mixing of the elastomer composition was done as a one-step process by using a horizontal sigma blade mixer at 65°C. All the ingredients except the curing agent and curing catalyst were blended for about 15 min. The mixing was continued for another 45 min under the same conditions, but this time in vacuum. After adding the curing agent (IPDI), the slurry was further mixed about 15 min in vacuum. The NCO/OH and Triol/Diol ratio were kept constant at values 1.1 and 0.054 in formulations, respectively. The weight percentage of the bond promoters in the composition was changed in the range of 0–4 wt % by an increment of 0.5 wt %. Carbon black concentration was kept at 10 wt % in all formulations.

The composite was composed of 10 wt % prepolymer, IPDI as a curing agent, AP as an oxidizer, crosslinking agent (triethanol amine), plasticizer (dioctyl adipate), bonding agent (TEPANOL), metallic fuel (aluminum), and burning rate modifier (Fe₂O₃). The NCO/OH ratio, triol/diol ra-

tio, and the solid loading of the composite propellant were all kept constant.¹⁰

All test specimens were prepared by using the respective elastomer compositions cured at 65°C for 7 days. To determine the adhesive properties of elastomer, the elastomer had to be cured on metal specimens for 4 h at 65°C until they reached a tacky state, then prepared composite was cast onto the precured elastomer and they were cocured for 7 days at 65°C.

Methods of Testing

Testing of Adhesive Properties of Elastomer

The bonding performance of the metal-elastomer-composite (MEC) system under tensile stresses was tested with 10-mm/min operational crosshead speed and the type of failure was determined by using the MEC tensile test.¹¹ The bonding characteristics between elastomer and composite and the type of failure in the system were determined under 90° peel forces by using the MEC peel test with 10 mm/min.¹¹

The adhesion ability of elastomer to the metal case was characterized by the peel test. The peel test was performed under peel forces with a load at 90° and a constant crosshead speed of 100 mm/min. The average peel strength and type of failure were analyzed by T-peel test which is defined as a test method for determining the relative peel resistance of adhesive bonds between flexible adherents by means of a T-type specimen.¹² To determine the adhesion character of elastomer to the metal case, the shear test was performed. The shear test is used to determine the comparative shear strengths of adhesives for bonding metals when tested on a standard specimen and under specified conditions of preparation and testing.¹³ The load at the rate of 1 mm/min was applied to the sample within the plane of the adhesive layer. The two adherents were pulled in opposite directions and the shear strength of the adhesive layer and the nature of the failure were analyzed. The Instron Testing Machine, Model 1185, with software produced by Hewlett Packard was used for adhesive characterization tests.

Testing of Mechanical Properties of Elastomer

The mechanical characterization of elastomer and qualitative comparison of different elastomer compositions were determined by using the tensile test method, in the form of standard dumbbell-shaped test specimens.¹⁴ The load was ap-

plied perpendicularly to the plane of the specimen with 50-mm/min crosshead speed which is within the test range of elastomers in testing standards. Tensile strength at the yield point, percent elongation at break and modulus of elasticity (initial modulus), were obtained in tensile tests. The Instron Testing Machine was also used for adhesive characterization tests.

Hardness Test

Hardness is a composite property combining concepts of resistance to effects such as penetration, scratching, and marring.¹⁵ The hardness values of the composite placed between two elastomer layers in the MEC-tensile test specimen was determined to characterize and to analyze changes in microstructure of the polymer matrix. A KORI Shore A type rubber hardness tester was used to measure the hardness values of the matrixes.¹⁶ The hardness value was based on the resistance to penetration by an indenter pressed into the specimen under a constant load. Hardness values were read after waiting for a few seconds, when the indicated value stabilized.

Scanning Electron Microscopy (SEM) Measurements

SEM images of the sample were taken on a JEOL-JXA 840 A SEM instrument operating at 20 kV and with a 40 Å point-to-point resolution. Typically, SEM pictures of samples were taken from the freshly cut surfaces at three different magnifications to obtain information about the sample in general.

RESULTS AND DISCUSSION

The bonding performance of the aziridine-type bond promoters BITA and MAPO in an HTPB-based polyurethane elastomer was studied in terms of adhesive and mechanical properties. A series of HTPB-based elastomeric liners of otherwise the same composition were prepared by using either MAPO or BITA as bond promoters in various concentrations up to 4.0% for a comparative investigation. For the adhesive properties of elastomer, the performance of the bonds at the interfaces of the metal case-elastomer-composite system were analyzed under tensile and peel forces, and the bonds at the interface of the binary elastomer-metal case were examined under peel

and shear forces. The elastomer compositions were tested for their mechanical properties by using tensile and hardness tests. The structural properties of the composite material that was placed between two elastomeric layers in the metal case-elastomer-composite systems were also obtained by using the hardness test. This method was also used to determine the failure type in the system under forces.

Bonding Mechanism

A major pathway of interaction of aziridine-based bond promoters in elastomeric liner or composite propellant is the ring-opening reaction of the aziridine with hydroxyl groups of HTPB binder.¹⁷ The latter also reacts with the isocyanate groups of curing agents forming the crosslinked polyurethane elastomer. This possible mode of action of aziridine groups also involves crosslinking of the polymer network at the elastomer-composite interface through reaction with the free hydroxyl groups in the matrix. Hydrogen bonding between aziridine converted into a crosslinked polymeric form in elastomer and AP in the composite matrix at the interface will also enhance the elastomer-composite interfacial force.

When the elastomeric material comes into contact with the surface of metal, it interacts on the metal surface by the help of atomic forces on the surface.¹⁸ The adhesive ability of elastomer containing bond promoter can be improved by the pretreatment of the metal surface with a primer organic isocyanate. Thus, the isocyanates react both with the surface hydroxyl groups and imino groups generated upon ring opening of aziridine-type bond promoters.¹⁹

Effect of Bond Promoters on the Adhesive Properties of Elastomer with Composite Matrix

The interfacial bond characteristics between the elastomer and composite were studied by using two structurally different bond promoters in various concentrations up to 4% in an HTPB-based polyurethane elastomer, which otherwise has a constant composition. The effect of bond promoter concentration on composite matrix-elastomer adhesion under tensile and peel stresses is shown in Figures 2 and 3, respectively. Both the tensile and peel strengths at the elastomer-composite interface increased first with the increasing percentage of bond promoter, reached a maximum at 1 wt %, and then started to decrease. Thus, 1 wt % of

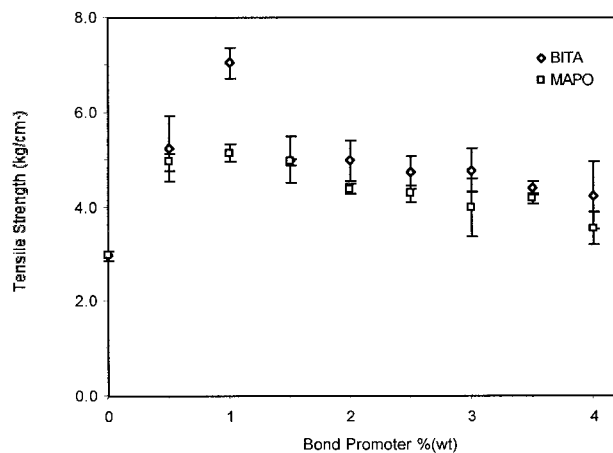


Figure 2 The effect of bond promoter concentration on composite matrix–elastomer adhesion under tensile stresses.

the bond promoter in the elastomer could be considered to be the optimum concentration for achieving the best adhesion of the elastomeric liner to the composite propellant. Further addition of the bond promoter beyond this value reduces the bonding performance of the elastomer. The first increase and then decrease observed in the adhesive properties of elastomer with composite matrix are the result of several reasons. The bond promoters could migrate into the composite and come into contact with the AP particles at the composite interface as soon as the composite is cast. Up to a certain amount of bond promoter in elastomer, the formation of rearrangement products such as oxazoline in the elastomer was slow

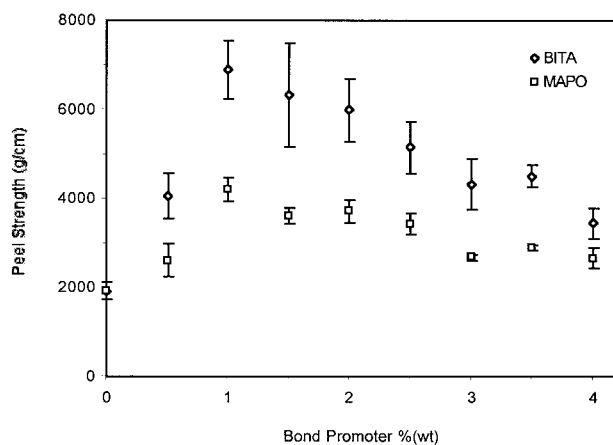


Figure 3 The effect of bond promoter concentration on composite matrix–elastomer adhesion under peel stresses.

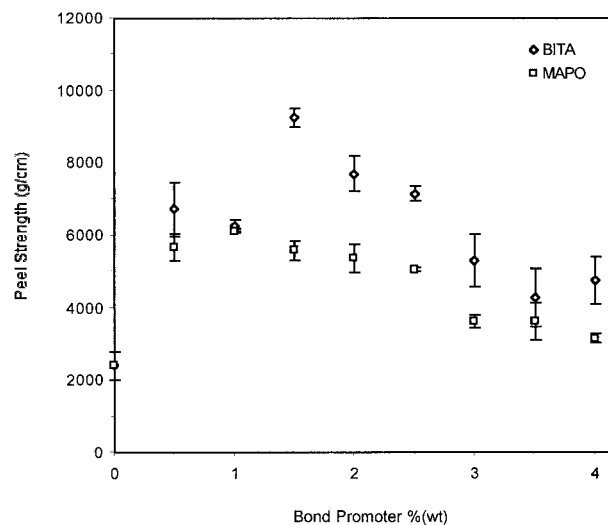


Figure 4 The effect of bond promoter concentration on metal–elastomer adhesion under peel stresses.

enough to allow significant migration and reaction of aziridine at the composite interface. But, in the presence of the excess amount of bond promoter, the rearrangement of some of the aziridines could lower the bonding performance of the system because of the lack of hydrogen bonds between the composite matrix and elastomer. This general trend was more noticeable for BITA compared with MAPO, as expected from the suitable spatial arrangement of three aziridine groups in BITA.

Effect of Bond Promoters on Adhesive Properties of Elastomer with Metal Surfaces

As shown in Figure 4, increasing concentrations of bond promoter in elastomer cause the peel strength of the bonds to increase until reaching a maximum. Between the bond promoter concentrations of 1.5–5 wt %, the peel resistances of elastomer compositions with BITA are remarkably stronger than those of elastomer compositions with MAPO. The specimens were also analyzed under shear stresses, and these test results are given in Figure 5. In this case, a maximum stress is obtained at 1.5 wt % bond promoter.

Effect of Bond Promoters on the Nature of Failure in the Elastomer–Composite System

In addition to the analysis of the changes in bond strength between elastomer and composite, the nature of failure was also investigated, and the

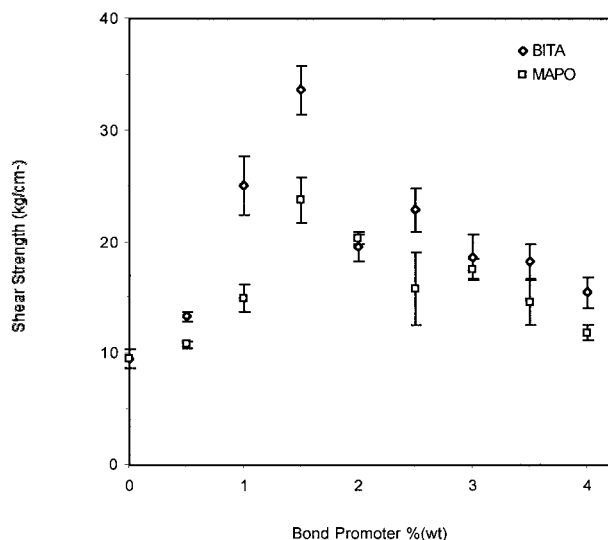


Figure 5 The effect of bond promoter concentration on metal–elastomer adhesion under shear stresses.

results are summarized in Table I. It is not surprising that the failure is usually on the composite side of the interface because the tensile strength and elongation of the composite were considerably lower than those of the elastomer. This indicates that the failure is caused by a weak boundary layer of material at or very close to the interface in the composite site. During curing, a weak layer in the composite matrix near the elastomer interface is formed. This is caused by the migration of isocyanate from the composite into the precured elastomer. Bond promoters in elas-

tomers prevent this migration when used in larger amounts. Some amount of bond promoter in elastomer disperses into the composite matrix and accelerates the matrix curing by slowing down IPDI migration out of the composite matrix.²⁰ This increases the crosslinking of the composite matrix binder. Moreover, the presence of bond promoter in elastomer enhances the composite–elastomer adhesion by restoring the appropriate mechanical properties of the composite matrix near the interface where the bond promoter reacts with the surface of AP.

The interfacial layer characteristics between elastomer and composite were examined with SEM. An SEM image is given in Figure 6, which indicates that two layers were strongly drifted into each other. Perfect interfacial features without any deformation and void formation are seen between these two polymer matrixes.

Generally for the bond promoters of aziridine type, hydrogen bonding occurs at the AP surface in actual composite interface and forms a tough thin layer surrounding each AP particle. This toughly bonded high modulus layer, which is clearly seen in the SEM image (Fig. 6), improves the adhesion properties of the composite–elastomer system up to a certain point. This suggests that the actual interface is commonly composed of a thin film of composite matrix binder on the composite side and a likewise thin polymer layer on the elastomer side leading to the cohesive failure.²¹

When a load was applied to the interfacial system as peel stresses, vacuoles formed around the

Table I The Effect of Bond Promoter Concentrations on Type of Failure in Elastomer–Composite Systems Under Stresses

Bond Promoter (wt %)	BITA		MAPO	
	Type of Failure Under Tensile Stresses	Type of Failure Under Peel Stresses	Type of Failure Under Tensile Stresses	Type of Failure Under Peel Stresses
0	CFI	AF	CFI	AF
0.5	CFI	CFI	CFI	AF
1	CFP	CFP	CFP	CFP
1.5	CFP	CFP	CFP	CFI
2	CFP	CFI	CFP	CFI
2.5	CFI	CFI	AF	AF
3	CFI	CFI	CFI	AF
3.5	CFI	CFI	AF	AF
4	CFI	CFI	AF	AF

AF, adhesive failure; CFI, cohesive failure at interface; CFP, cohesive failure at propellant.

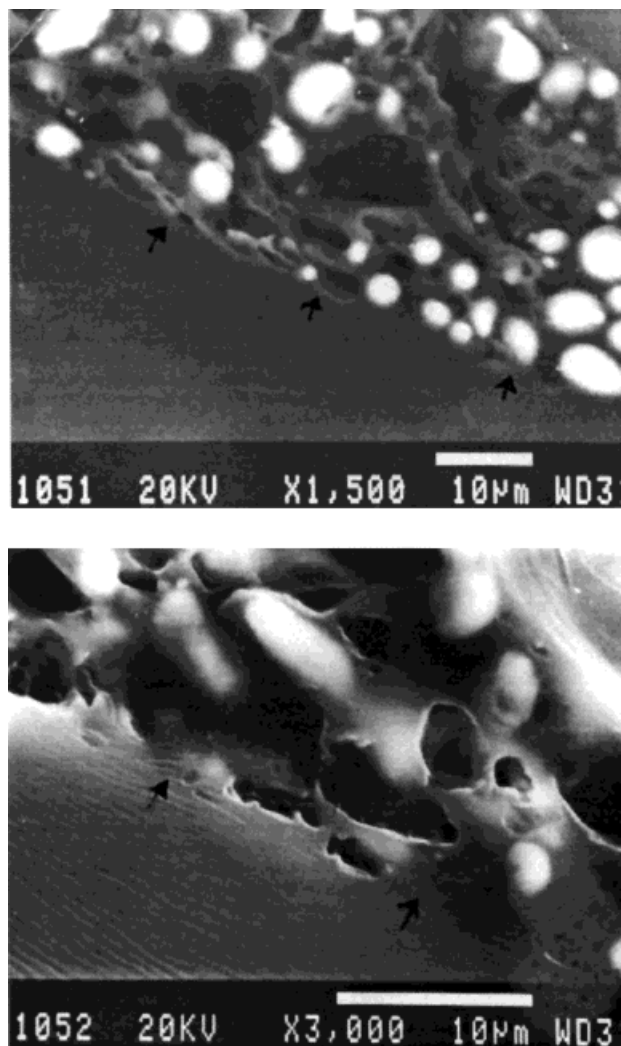


Figure 6 SEM images showing the interfacial layer between composite matrix and elastomeric liner. The light contrasted spherical grains with 1.5–10- μm particle size are aluminum-rich particles. Gray contrasted small particles with 5- μm diameter and large grains with 150- μm particle sizes are chlorine-rich particles of AP. The binder in gray contrast forms a drifted, composite structure with the light contrasted spherical aluminum and AP grains in the propellant matrix.

AP particles at the interface during dewetting.⁹ Elastomer with bond promoter surrounds the AP particles at the interface and under stresses and continues sharing the load in the system until the dewetting is completed.

Effect of Bond Promoters on Mechanical Properties of Elastomer

The effect of bond promoter concentration on mechanical properties of the elastomeric liner in

terms of tensile strength, elongation, initial modulus, and hardness values is summarized in Table II. If the tensile strength of the elastomer is not high enough, the composite will not be held on the metal surface under high stresses. Insufficient elongation does not prevent separation in the metal case-elastomer-composite system. However, elastomer compositions having high elongation values are easily deformed during storage and operation of the rocket motor. Also, neither low nor high modulus is suitable for elastomer in use. Elastomeric liner with low modulus causes excessive deformation in the composite grain because of the creep, and elastomer with high modulus has lower strain capability.

An excess addition of bond promoter to the elastomer system changes the major reaction pathway of the aziridine in the HTPB environment. Bond promoters increase polar groups in the elastomer concentration. These polar groups attract each other and form hydrogen bonds, so residual stress in the elastomer increases. The rearrangement of aziridines is also identified as another process in the system.¹⁷ The change in the ratio of aziridine structure in elastomer during curing gives some insight into the structure change of the polymer. Some of the polar groups are still free to form strong hydrogen bonds, although to a lesser extent. The mobility of the free groups in the system is reduced because of the presence of strong hydrogen bonding.

Comparison of Bond Promoters BITA and MAPO

Elastomer compositions with bond promoter BITA had better bonding performance with composite and metal case and better mechanical properties compared with those with MAPO. Because of its lower molecular weight than BITA, there are more repeating reactive groups of MAPO at the same bond promoter level in the elastomer compositions. However, adhesive and mechanical performance of BITA under shear, peel, and tensile forces are better, or at least the same as, MAPO. This can be explained as follows:

- Geometrical configuration: BITA has three aziridine groups arranged in trigonal planar configuration, whereas the three aziridine groups in MAPO are arranged in a trigonal pyramidal shape (Fig. 1). Functional groups on planar structures can react readily to form crosslinks and hydrogen bonds with other groups.

Table II The Effect of Bond Promoter Concentrations on Mechanical Properties and Hardness of Elastomer

Bond Promoter (wt %)	BITA				MAPO			
	Tensile Strength (MPa)	Elongation (%)	Initial Modulus (MPa)	Hardness (Shore A)	Tensile Strength (MPa)	Elongation (%)	Initial Modulus (MPa)	Hardness (Shore A)
0	0.848 ± 0.032	262 ± 13	0.238 ± 0.009	30.6 ± 0.89	0.848 ± 0.032	262 ± 13	0.238 ± 0.009	30.6 ± 0.89
0.5	1.337 ± 0.072	313 ± 26	0.425 ± 0.127	34.8 ± 0.84	1.035 ± 0.057	288 ± 9	0.394 ± 0.025	33.8 ± 0.45
1	1.928 ± 0.138	321 ± 25	0.785 ± 0.050	44.0 ± 1.41	1.127 ± 0.091	377 ± 22	0.380 ± 0.053	34.0 ± 2.00
1.5	2.103 ± 0.057	325 ± 16	0.828 ± 0.044	44.4 ± 1.67	1.249 ± 0.122	363 ± 16	0.393 ± 0.061	37.4 ± 1.95
2	1.813 ± 0.200	327 ± 33	0.834 ± 0.071	43.6 ± 1.67	1.560 ± 0.149	380 ± 9	0.620 ± 0.035	41.8 ± 1.48
2.5	1.883 ± 0.112	321 ± 6	0.801 ± 0.077	42.0 ± 1.41	1.901 ± 0.216	381 ± 11	0.700 ± 0.016	40.2 ± 1.10
3	1.663 ± 0.078	329 ± 14	0.617 ± 0.073	40.4 ± 0.55	1.457 ± 0.056	446 ± 32	0.400 ± 0.036	39.6 ± 0.89
3.5	1.641 ± 0.077	372 ± 40	0.492 ± 0.013	38.4 ± 2.61	1.222 ± 0.121	473 ± 21	0.372 ± 0.047	37.6 ± 1.52
4	1.332 ± 0.132	440 ± 35	0.241 ± 0.053	33.4 ± 0.89	1.210 ± 0.162	479 ± 16	0.225 ± 0.023	35.4 ± 0.89

- The electronegative oxygen atoms in the bond promoter molecule can play an important role too. Although MAPO contains one oxygen atom (P=O double bond) in its structure, BITA has three carbonyl groups (C=O double bond); therefore, the possibility of hydrogen bond formation is higher for BITA than that for MAPO. Moreover, the C=O double bond is more polarized than the P=O double bond, because of higher electronegativity difference between carbon and oxygen than that between phosphorus and oxygen. As a result, BITA carbonyl groups, more polar and spatially more available, can make stronger bonds to hydrogen atoms of AP than the one P=O group of MAPO does.

CONCLUSION

In most of the adhesive systems, the highest stresses and strains usually occur at discontinuities of the composite–elastomer and elastomer–metal interfaces. Therefore, the adhesive and mechanical characteristics of an elastomer which act as a binding material between composite and metal keep its importance as a design parameter. The configurations of composite grain, elastomer, and grain support are designed in such a way that there will be no failure because of excessive stresses. The characteristics of a viscoelastic material can lie in a wide range if sufficient modifiers and additives are used.

An improvement in the adhesion characteristics of elastomeric liner was achieved by optimizing the amount of bond promoter used in the elastomer compositions. Although elastomer compositions with 1.0, 1.5, and 2.0 wt % BITA are preferable because of their high tensile and peel strengths, all the elastomer compositions with bond promoters BITA and compositions with 1.0, 1.5, and 2.0 wt % MAPO seem to be sufficient for adhesive systems. The mode of failure between the composite and the elastomer for all the systems studied are all acceptable. In other words, the interfacial adhesive strength of these compositions are higher than the cohesive strength of the composite matrix.

The adhesive ability of elastomer with metal was also highly improved by the addition of aziridine-type bond promoters to the elastomer. Although the failure type under peel and shear forces is always cohesive from elastomer as desired, elastomer compositions with bond promoter

quantities of 1.0, 1.5, 2.0, and 2.5 wt % have better strength values than the others.

All the elastomer compositions studied, having mechanical properties greater than those of HTPB-based composite propellant (tensile strength of nearly 6 kg/cm² and 70% elongation), are suitable for adhesive systems. However, the related properties of elastomers with 1.0, 1.5, 2.0, and 2.5 wt % BITA are better than that of the others. They have higher tensile strengths with sufficient elongation values. Also, an elastomer composition with 2.5 wt % MAPO has high tensile strength, whereas the same strength value can be obtained by using BITA in lower quantities. Therefore, BITA can be preferred as bond promoter over MAPO in elastomeric compositions.

Taking this study as a guide, it can be concluded that elastomer compositions with bond promoter BITA give better bonding performance between the composite-metal system and better mechanical properties when compared with the elastomers with bond promoter MAPO. The best results are obtained by using the optimum quantities of bond promoter BITA such as 1.0 and 1.5 wt % loadings in the elastomer compositions.

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