

# Characterization Studies on Aging Properties of Acetyl Ferrocene Containing HTPB-Based Elastomers

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Received 12 April 2005; accepted 11 December 2005

DOI 10.1002/app.23918

Published online in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** In composite solid propellants, low-molecular-weight species such as burning rate catalysts, plasticizer, etc. which migrate into liner and thermal insulation layers during curing and storage invariably result in poor mechanical and ballistic properties of the propellants. In the present study, the migration of the burning rate catalyst, acetyl ferrocene, was investigated spectrophotometrically (UV-visible) by evaluating the extent of hindrance to such migration after applying a barrier (liner) of various crosslink densities between the additive (HTPB-TDI-plasticizer-acetyl ferrocene) and nonadditive (HTPB-TDI) gumstocks replicating

the propellant and insulating layer, respectively. Enhancing the crosslink densities of liner via a trifunctional aziridine crosslinking agent inhibited migration. The aging of additive gumstock was done at 60°C and its mechanical properties and extent of acetyl ferrocene migration were also evaluated and analyzed. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 2538–2545, 2006

**Key words:** hydroxyl-terminated polybutadiene; burning rate catalyst; acetyl ferrocene; aging; migration; UV-visible spectrophotometer

## INTRODUCTION

Composite propellants are made of a polymeric matrix, additive with a solid powder oxidizer, and possibly a metal powder that plays the role of a secondary fuel component. Polymeric matrix is the main element of composite propellants. Hydroxyl-terminated polybutadiene (HTPB) liquid prepolymers play an important role in the manufacture of composite solid propellants for rocket motors on account of their specific properties and desirable mechanical properties of the grain.<sup>1</sup> These binders allow a high oxidizer to fuel ratio and they lead to the considerable development of composite propellants in large case-bonded grains that were impossible to manufacture with other types of propellants. HTPB has long been one of the most commonly used polymers in composite solid propellants. Its role in composite solid propellants is mainly twofold, one of which is serving as a binder material for physical accommodation of different propellant ingredients (such as oxidizer, metal particles, and burn-rate modifiers) in its matrix. The other is serving as a fuel constituent to release fuel-rich gaseous products when decomposed and pyrolyzed.<sup>2–4</sup>

During storage and transportation, the solid rocket propellants are subjected to several loading conditions such as humidity, temperature, vibration, and shocks.

They suffer both from chemical and physical aging, which consequently lead to changes in their mechanical properties. One of the most important parameters that determine the shelf life of the propellant system is the migration of low-molecular-weight species from the propellant into insulation layer of the rocket. The migration that occurs in rocket motors is a result of concentration differences in mobile species across bi-material interfaces. If the mobile species are not chemically bonded to a substrate material, then they eventually migrate toward lower concentrations. The migration of low-molecular-weight additives such as plasticizers and ferrocene derivatives from the propellant into the insulation during curing and aging processes is of considerable scientific and industrial importance. They migrate through the cured propellant into the liner and onto the surface of the propellant grain. This can lead to localized high burning rates in the motor, causing pressure fluctuations which may lead to motor failure.<sup>5–9</sup>

The industrial importance arises because of the fact that migration of mobile species changes the main designed performance characteristics of the rocket motor considerably. Migration is expected to affect the following: mechanical properties of the propellant and insulation, bonding between the propellant and insulation, and burning rate characteristics.<sup>10,11</sup> Therefore, it is important to characterize, eliminate, or inhibit the migration in such systems. Subramanian<sup>12</sup> has reported the thermal and aging studies on vinyl ferrocene grafted HTPB polymeric binder to evaluate its

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performance as a propellant binder. Swett<sup>13</sup> and Venkatesan et al.<sup>14</sup> have studied that the migration behavior of both plasticizer and burning-rate catalysts are followed mainly by using GC-MS and HPLC chromatographic techniques. In the present study, the main interest is certainly the aging effects of the elastomeric system on thermal, mechanical properties, and migration behavior of acetyl ferrocene, which serves as burning rate catalyst. We also concentrate on recognition of acetyl ferrocene migration qualitatively throughout HTPB-based elastomeric samples. The characterization studies and migration scheme of acetyl ferrocene contained in HTPB-based elastomers were achieved by employing different analytical techniques such as UV-visible, thermal, and mechanical analysis methods.

## EXPERIMENTAL

### Materials

HTPB was provided from ARCO chemical Co., Philadelphia, PA. The number-average molecular weight and hydroxyl value of HTPB are 2975 g/mol and 0.60–0.70 mmol/g, respectively. Dioctyl adipate (DOA), acetyl ferrocene, and benzene were supplied from ACROS organics, BVBA/SPRL, Belgium. Toluene diisocyanate (TDI; Fluka AG, Germany), trifunctional aziridine (crosslinker AST D45+, KIMETSAN, Turkey) were all used as purchased.

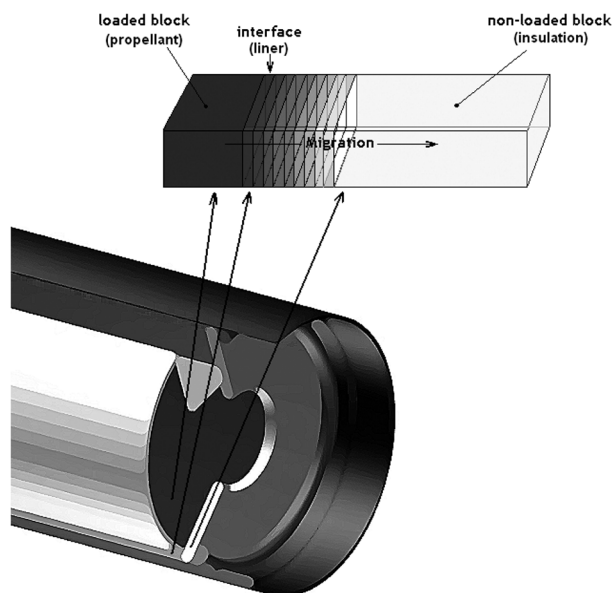
### Sample preparation

#### Preparation of HTPB-based elastomer specimens

*R*-value is an important design parameter for obtaining the chemical crosslink in propellant grain's matrix. The urethane networks composed of HTPB, TDI, and DOA in different equiv. of [NCO]/equiv. of [OH] (= *R*) values ranging from 0.85 to 1.10 are prepared by homogeneous blending of the HTPB with predetermined amounts of DOA followed by the addition of required amount of TDI with continuous stirring for 20 min to get a homogeneous slurry mass. Mixture is then cast into teflon molds and cured at 60°C over various periods of time for determining the curing period of the prepared specimens.

#### Preparation of test specimens for migration studies

The specimens were prepared at three stages to simulate the original location of the rocket in which the migration process takes place. Prepared samples composed of three integrated, adjacent blocks (non-additive/interlayer/with additive) of elastomer. These three blocks correspond to the insulation/liner/propellant parts, respectively, in the rocket applications (Fig. 1). Mixing size is fixed as 10 g and 20 g for



**Figure 1** Illustration of propellant segments in which migration takes place.

preparation of interlayer and additive blocks, respectively, and 50 g for nonadditive part. The aging period for nonadditive part was kept long enough to observe migration behavior of acetyl ferrocene in the system. For all triblock specimens, following experimental preparation steps were used. At first step, the nonadditive block, which is only composed of HTPB and TDI, is cured at 60°C. This block is then transferred into another larger mold and besides an interlayer part is cured on one of the surface of the previously prepared block. Trifunctional aziridine of 7% by weight as a crosslinker is used for the preparation of the interlayer part. Finally, the third layer is cast on the surface of this interlayer. Third block (additive block) is composed of HTPB, TDI, plasticizer (DOA), and burning rate catalyst (acetyl ferrocene). In mixing process of the additive block, DOA and acetyl ferrocene ratio are kept at around 2.85% and 0.83% by mass in composition, respectively. Then prepared samples are aged at 60°C with varying time.

### Studies/characterization

#### Optimization studies of curing time

For the specimens with *R* = 0.95, curing processes are carried out for various curing periods from 1 to 7 days. Change in the mechanical properties of elastomer specimens was investigated by conventional uniaxial testing system for determination of effective curing time.

#### Tensile tests

Tensile tests of the samples are carried out at room temperature. Stress/strain properties of all samples

are tested on Lloyd LS 500 computer-controlled tensile testing machine. The gauge length was fixed to 30.0 mm. Load cell (100 N) was used for each test. The crosshead speed is altered to 50.0 mm/min. The data presented are the average of at least six measurements.

#### Differential scanning calorimetry

The thermal properties of samples are examined by using differential scanning calorimeter unit of TA instrument of DSC 910S (V4.1). Differential scanning calorimetry (DSC) thermograms were recorded in nitrogen atmosphere between  $-120$  and  $400^{\circ}\text{C}$ . The samples are cooled at a rate of  $10^{\circ}\text{C}/\text{min}$  using liquid nitrogen to the required temperature and then heated at a rate of  $10^{\circ}\text{C}/\text{min}$ . The weights of the samples used are in the range of 5–10 mg. Decomposition and glass transition temperatures of the selected specimens were recorded on the DSC thermogram.

#### Characterization of migration of acetyl ferrocene with UV-vis spectrophotometric method

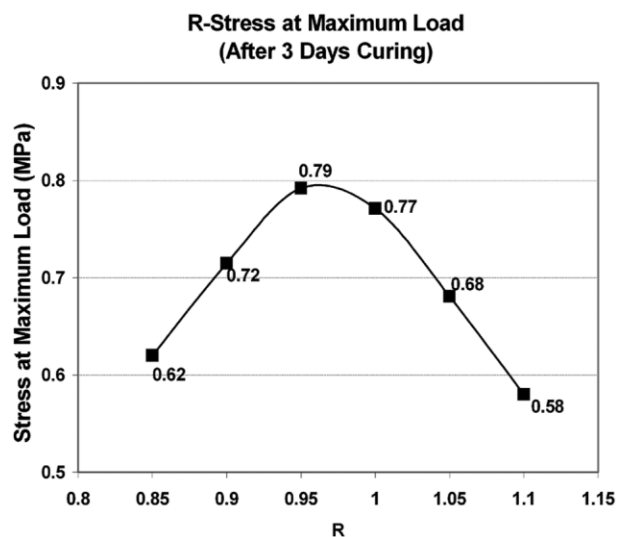
Prepared triblock samples are cut into slices as shown in Figure 1. These slices were chopped by means of a blade and then the desired amount of sample was weighed for extraction. For extracting acetyl ferrocene from the slices, benzene was used as the solvent. Benzene penetrates and swells the rubbery structure remarkably and is also a good solvent for acetyl ferrocene extraction. Benzene is also feasible because of its higher boiling temperature for quantitative studies at room temperature conditions. Slices obtained from the blade cutting are weighed to calculate the  $\text{mg}/\text{g}_{(\text{slice})}$  values for the plot of acetyl ferrocene concentration versus migration distance.

For every sample extraction process took 16 h with refreshing extractor for several times. The UV spectrum for acetyl ferrocene shows maxima at 293.5, 336, and 455 nm.<sup>15</sup> Acetyl ferrocene is extracted from each chopped sample set by using equivolume extractor (benzene). Finally, 455 nm is selected and UV-vis tests are accomplished by using the UV-vis spectrophotometer unit of a Shimadzu instrument of UV-1201 at the same wavelength. During extraction of acetyl ferrocene, DOA is also extracted from samples. Since DOA has no absorption at 455 nm at a significant level, one can assume that there is no contribution to the total absorbance of extracted eluent and the benzene solvent employed as the reference solvent in UV-vis measurements.

## RESULTS AND DISCUSSION

### Mechanical test results of HTPB-based elastomeric system

The specimens with different  $R$ -values from 0.85 to 1.1 with different curing times were prepared to deter-



**Figure 2** Variation of stress at maximum loads for specimens with different  $R$ -values at constant curing time (3 days).

mine the compositions and the curing process parameters of the system. Tensile values of the specimens are plotted against different  $R$ -values for 3 days of curing period in Figure 2. It is seen that the ultimate tensile strength values (UTS) increase with increasing  $R$ -values up to 0.95, and then start to decrease. In our system, the best mechanical properties are obtained at  $R = 0.95$ , which is higher than the expected value of  $R = 0.85$ . This can be attributed to the presence of humidity in reaction medium of isocyanate with polyol. Isocyanate readily reacts with water appearing from atmospheric humidity in the time of curing. This will naturally increase the rate of isocyanate consumption in urethane formation reaction and thus result in a higher  $R$ -value than the expected one. The formulations that contain  $-\text{NCO}$  groups in excess over the stoichiometric requirement are likely to undergo the following competing reactions: isocyanate dimerization, biuret and allophanate formation; allophanate formation is most likely to happen under the reaction conditions of excess  $-\text{NCO}$  (HTPB-TDI).<sup>16</sup> From Figure 3, it can be observed that curing periods after 3 days were not actually so effective on the mechanical properties when compared to the  $R$ -value (in Fig. 2). In our experimental condition, it was not possible to reach the maximum mechanical strength (0.79 MPa), which is obtained when the specimen is prepared at  $R$ -values lower or higher than 0.95, although all the samples were cured during the same time period of 3 days. Only those samples prepared at an  $R$ -value of 0.95 could reach the desired mechanical strength, which enhanced our choice of selecting an  $R$ -value of 0.95.

Another set of experiments were performed with the samples prepared again at an  $R$ -value of 0.95, this

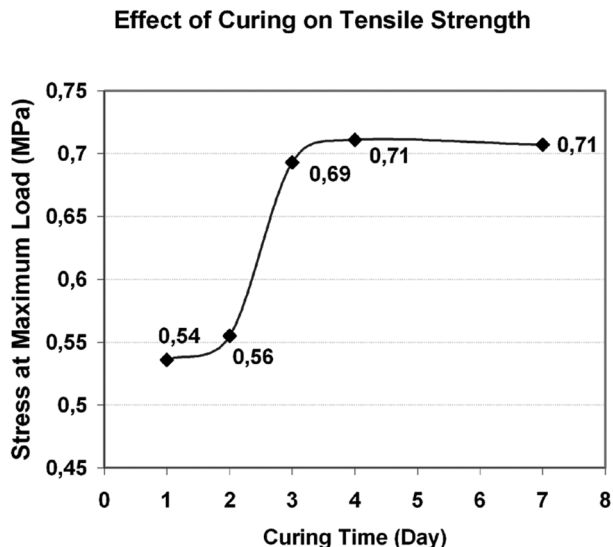


Figure 3 Variation of stress at maximum loads with curing time ( $R = 0.95$ ).

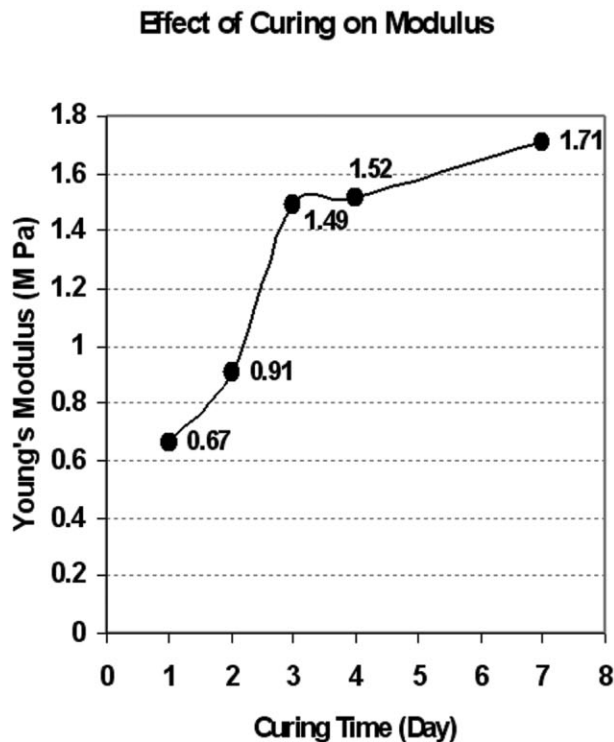
time only increasing the curing period. The obtained results are displayed in Figures 3, and 4(a,b). UTS and modulus values increase with increasing curing time up to 3 days, after which they reach a plateau, and percentage strain at break values show a decrease with increasing curing period up to 3 days, after which a plateau is obtained. Similar behavior has also been reported in literature.<sup>16,17</sup>

**Aging studies**

Degradation of the solid propellant is one of the most important factors limiting the lifetime of rocket motors. An accurate prediction of the aging behavior of the propellant is therefore essential for a safe and cost-effective use of propellant systems.<sup>12,18</sup> Aging procedure is performed at 60°C for the additive samples prepared at an  $R$ -value of 0.95 [Figs. 5, 6(a), and 6(b)]. Mechanical properties are determined after various specified aging periods at 60°C. Furthermore, a number of tests have been performed to identify the effect of aging on mechanical properties such as stress at maximum load, stress at break, and modulus. The rapid decrease in mechanical properties with increasing aging time is observed, which yields an increase in UTS and a decrease in strain at break values, most probably due to the increase in crosslink densities. During aging, strain capacities are observed to be more affected with aging time than maximum stress is. The modulus values did also increase with aging by the same reason. From the results of these tests, it can be concluded that aging process causes significant changes in mechanical properties of HTPB elastomeric system.

**DSC studies**

It was reported in literature<sup>19,20</sup> that for HTPB resin, there are two major possible degradation stages with indistinct separation. The temperature of maximum heat evolution is 370°C for the first degradation stage



**Effect of Curing on Elongation**

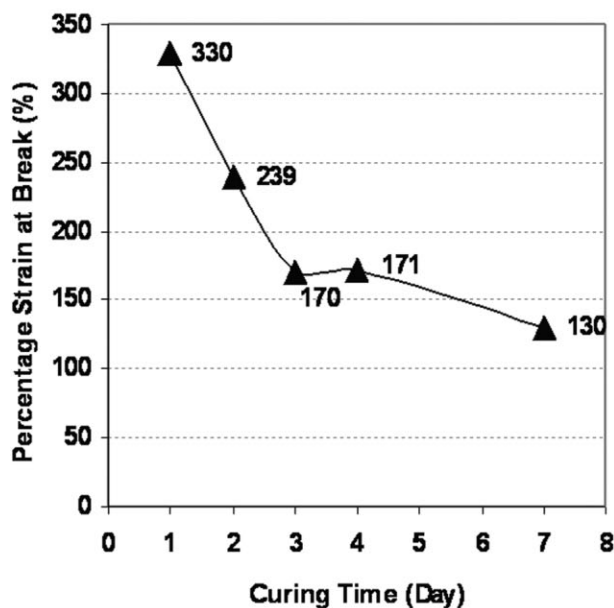


Figure 4 (a) Variation of modulus values with curing at constant  $R$  ( $= 0.95$ ). (b) Variation of percentage strain at break values with curing at constant  $R$  ( $= 0.95$ ).



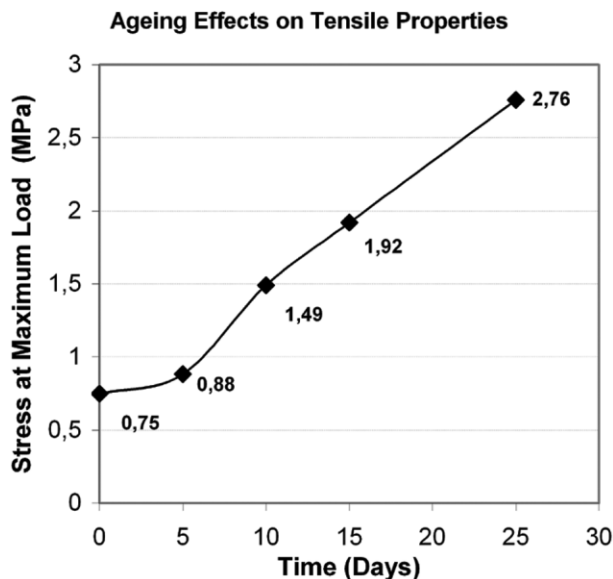


Figure 5 Effect of aging at 60°C on the tensile strengths of cured additive gum-stock for  $R = 0.95$ .

and at around 457°C for the second degradation stage. In our work, it was not possible to record the DSC traces beyond 400°C because of the working temperature limitations related with the DSC equipment. For that reason only the first decomposition transitions are observed at about 364°C. It is also clear from the DSC trace of nonadditive HTPB-based elastomer in Figure 7;  $T_g$  transition is indicated at about -77°C, as expected.

As stated in literature,<sup>3,20</sup> the first stage is primarily depolymerization, urethane bond cleavage cyclization, and crosslinking, accompanied by partial decomposition of the cyclized products. The major gaseous products are produced from the volatilization of low-molecular-weight hydrocarbons such as 1,3-butadiene ( $C_4H_6$ ), cyclopentene, cyclohexadiene, and 4-vinylcyclohexane ( $C_8H_{12}$ ). The second stage primarily involves decomposition of the cyclized products; meanwhile depolymerization and cyclization reactions gradually disappear in the second stage. The mechanism of polybutadiene degradation as suggested in literature is illustrated in Figure 8.<sup>21</sup> Main-chain scission results in chains with radical ends, which can either undergo depolymerization (butadiene and vinylcyclohexene) or cyclization to "cyclized" polybutadiene. The net exothermicity in the peak at about 364°C indicates that the energy released from the formation of new bonds during crosslinking and cyclization of HTPB-based elastomer is greater than the energy absorbed for bond scission during depolymerization.

Trifunctional aziridine, which is used in the composition of a barrier part for migration test specimens, has very little influence on the glass transition temper-

ature. This may be due to the fact that the glass transition temperature is dependent more on the segmental motion of the polymer chains rather than on the concentration of crosslinking points.<sup>22</sup> The concentration of aziridine obtained from KIMETSAN was very low because only 10% by weight aziridine was dissolved in ether and 7% trifunctional aziridine by weight was used from this solution, thus causing a little in-

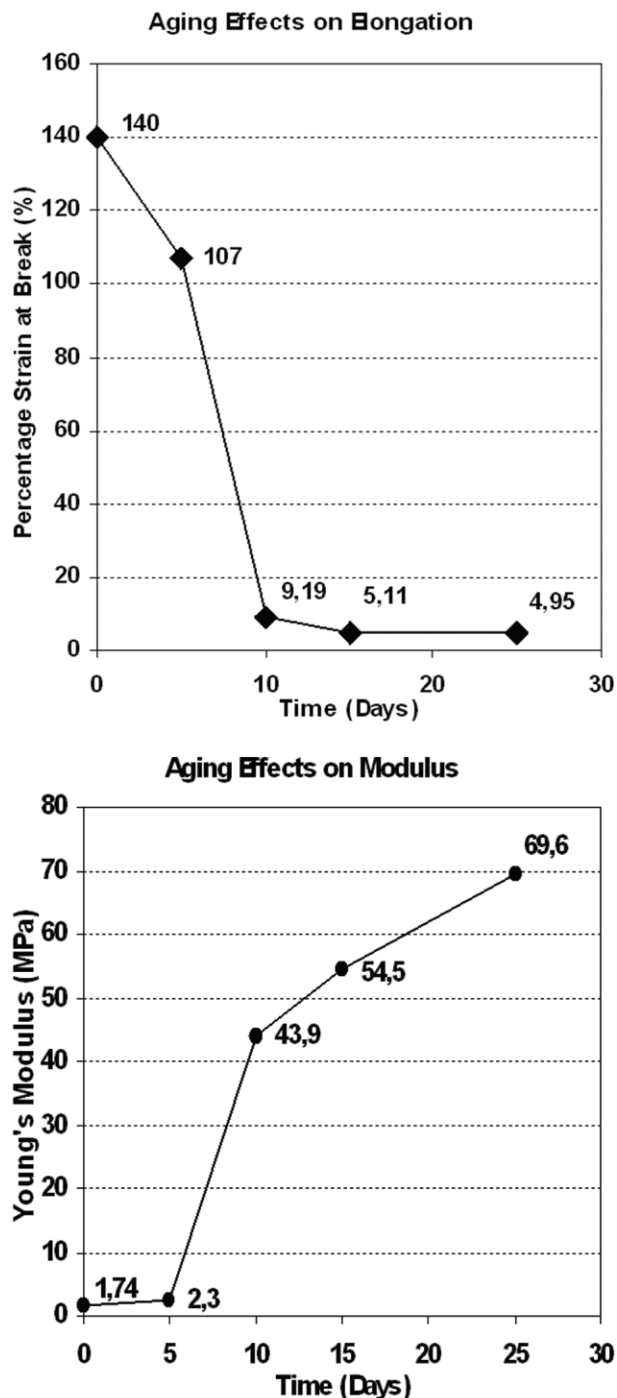
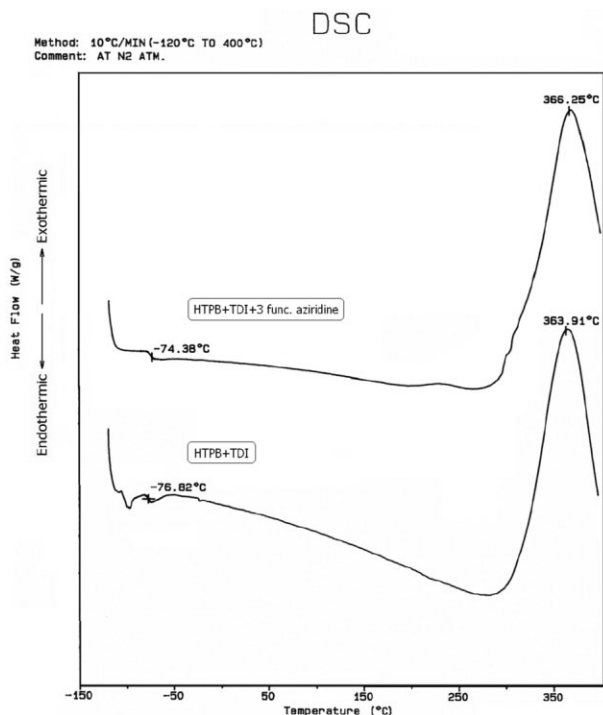


Figure 6 Effect of aging at 60°C on (a) elongation and (b) modulus of cured additive gum-stock for  $R = 0.95$ .



**Figure 7** DSC thermograms of nonadditive gum-stock (HTPB-TDI) and HTPB-TDI-trifunctional aziridine (method: rate of 10°C/min from -120 to 400°C in nitrogen atmosphere).

crease (only 2°C) in  $T_g$  of the elastomeric structure (Fig. 7). Although this amount was very small, it was sufficient enough to observe the inhibited acetyl ferrocene migration in the system.

Introducing acetyl ferrocene into the system causes significant changes in DSC thermograms of the elastomeric matrix. In the presence of acetyl ferrocene, first degradation temperature drastically decreases from temperature level of around 364°C to 315°C. This behavior can be attributed to a catalytic effect of ferrocene moiety on the degradation temperature of the system. Similar results have been reported<sup>12</sup> that trace-quantity of transition metals like iron promotes oxidative degradation of polymer. That catalytic effect is examined from the DSC thermogram of the sample with additive in Figure 9. In the DSC thermogram of sample with additive, additional one degradation peak is observed before the typical degradation peak of the butadiene elastomer at about 364°C. The transition at 315°C is much more intense than degradation transition of butadiene.

In Figure 9, it can be seen that there is a very weak exothermic peak at around 180°C, which can be attributed to oxygen vicinity from confined air in the sealed aluminum DSC sample packs during preparation of the samples. Despite the DSC tests performed at  $N_2$  atmosphere in our studies, some air may still remain in sealed sample packs and this will explain slight

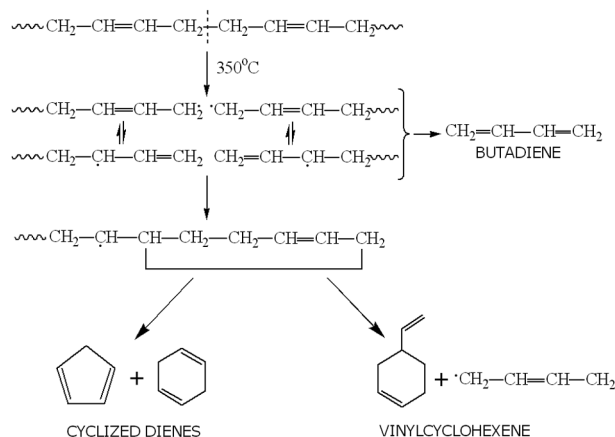
exothermicity in all of our thermograms. It was also reported in literature<sup>12</sup> that DSC thermograms of HTPB show some oxygen uptake peaks via hydroperoxidation and peroxidation reactions in air around 200°C.

The difference in peak areas of additive and nonadditive samples proves that the energy deployed from the system per gram substance increases with acetyl ferrocene (from 370 to 384.18 J/g) present in the decomposition range of 250–400°C. Acetyl ferrocene catalyst allows the HTPB-based solid fuel to burn faster and more completely, which would accelerate the burn rate of solid rocket fuels and develops the maximum amount of thrust possible from the fuel. It increases the energy of the system, thus yielding a much more desired product for the propellant system.<sup>12,23</sup>

Possible catalyzation effect of ferrocene derivative in stages of curing and aging may introduce additional crosslinks formed in the system. The increase in crosslink density explains the increase in heat of decomposition and decrease in degradation temperature.

### Migration studies

Composite propellants consist of polymeric binder (HTPB), curing agent, plasticizers, burning rate catalyst (ferrocene derivatives), and other additives. HTPB-based elastomers enable the migration of the plasticizer and ferrocene derivative, which are not bonded chemically in the propellant part during curing and storage (aging process) from propellant to insulation. To enhance the shelf life of the propellant, it is necessary to restrict the migration process. Acetyl ferrocene is used as a burning rate catalyst in the HTPB-based elastomeric matrix and is extracted with benzene from prepared samples for the migration studies. Concentrations of extracted eluent are evalu-



**Figure 8** Polybutadiene degradation mechanisms (cyclization reactions).

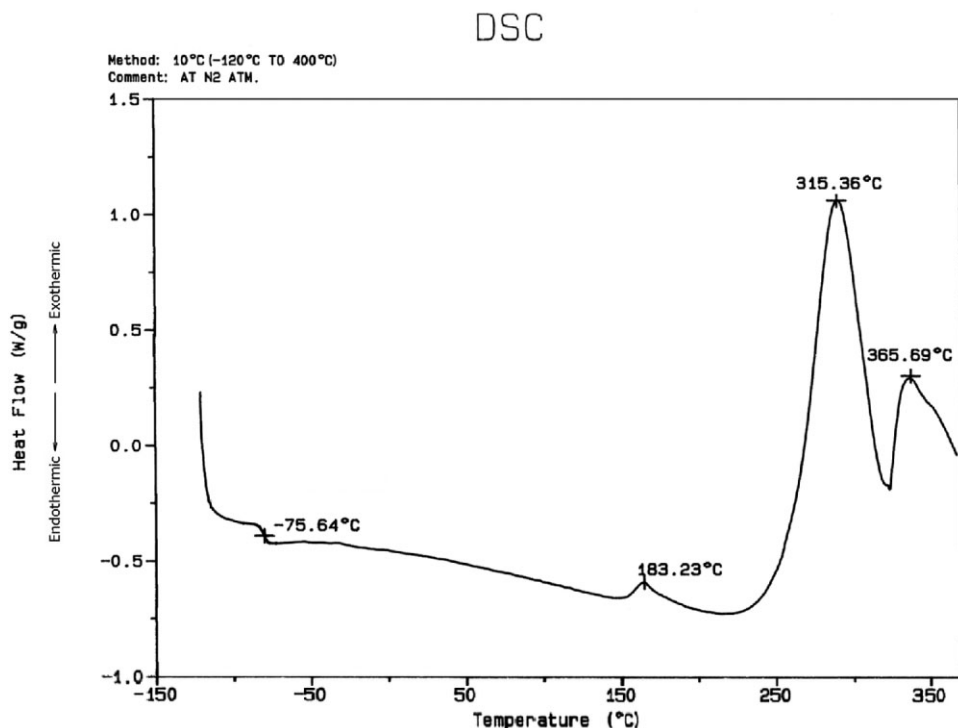


Figure 9 DSC thermogram of additive gum-stock (method: rate of 10°C/min from -120 to 400°C in nitrogen atmosphere).

ated from calibration curve previously developed. Then these data are converted to ppm scale, and finally mg/g<sub>(slice)</sub> data are calculated. Acetyl ferrocene analyses of these eluents are done by UV-vis spectrophotometry. In Figure 10, sample dimensions, starting point-zero, and approximate slice thickness are given. Data collected from the analysis of a nonaged sample are used to calculate acetyl ferrocene concentrations in slices of that sample and plotted against the migration distance of acetyl ferrocene in Figure 11.

Figure 11 shows that acetyl ferrocene concentration decreases gradually from the additive part to the non-additive part. It can be proposed that the migration of acetyl ferrocene from additive part to nonadditive part even takes place in curing process. Detailed results gained from extended works in characterization and

inhibiting acetyl ferrocene migration in HTPB-based elastomeric system and the effect of time and temperature on aging process were discussed in our previous study.<sup>24</sup>

To inhibit the migration of acetyl ferrocene and to increase the crosslinking density in the systems, a trifunctional aziridine as a crosslinker was added to the composition of the interlayer part between the additive and nonadditive blocks of specimens. At that

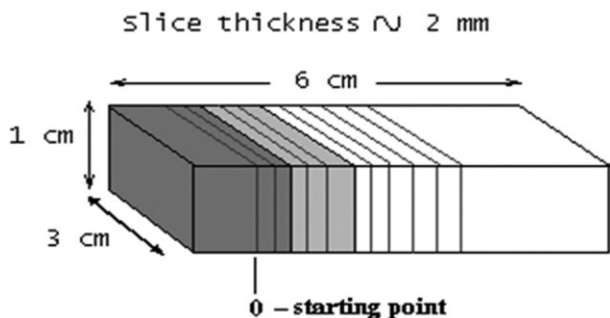


Figure 10 Sample dimensions, starting point-zero, and approximate slice thickness.

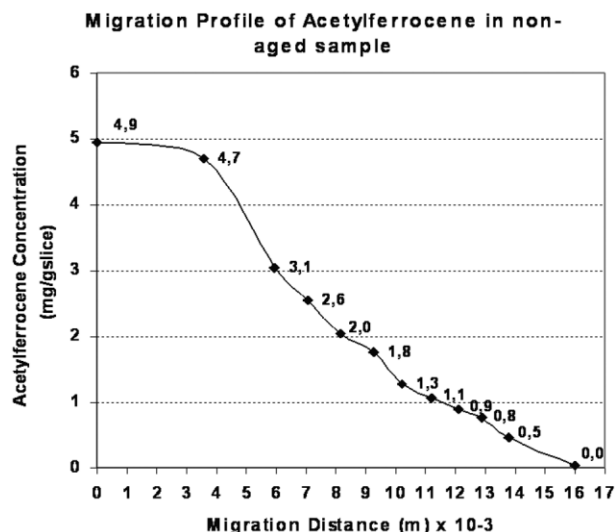
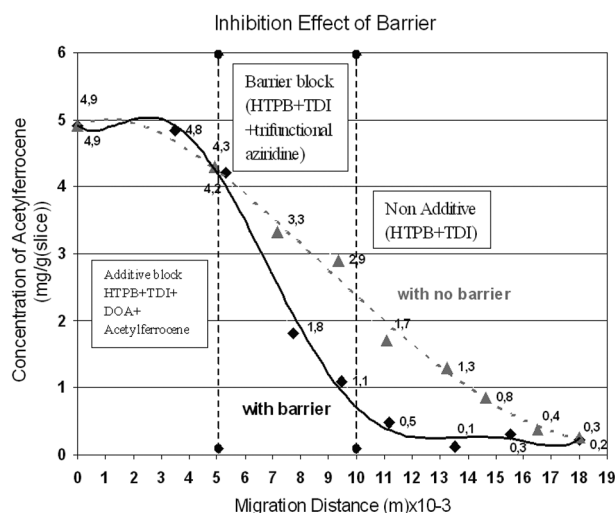


Figure 11 Migration profile of acetyl ferrocene in nonaged additive gum-stock cured for 3 days.



**Figure 12** The effect of trifunctional aziridine barrier on migration profile of acetyl ferrocene in aged additive gum-stock.

point, interlayer plays a role as a “barrier layer”. The parallel specimens prepared with and without barriers are analyzed with UV-vis spectrophotometer. Before testing, these two samples are kept at room temperature conditions for 10 days. Figure 12 shows the inhibition effect of the barrier layer on the migration profile of the system. At 11 mm, there is a drop of acetyl ferrocene concentrations from 1.7 (with no barriers) to 0.5 mg/g<sub>(slice)</sub> (with the barrier), indicating that large amount of acetyl ferrocene has migrated in the first case. From these results, it can be concluded that increasing crosslinking density in barrier structure inhibits the motion of low-molecular-weight (mobile) species as acetyl ferrocene in HTPB-based elastomeric matrix. Some further minimization studies have been accomplished and published previously.<sup>24</sup>

## CONCLUSIONS

The maximum UTS was obtained at an *R*-value of 0.95 for the HTPB-based elastomeric structure. The UTS, elongation at break, and modulus values reached a steady value during a 3-day curing time at *R*-value of 0.95. Mechanical properties are also determined after various specified aging periods at 60°C for the additive samples prepared at an *R*-value of 0.95. The rapid decay of mechanical properties with increasing aging time causes an increase in UTS and decrease in strain at break values due to the enhanced network structure. During aging, strain capacities are observed to be more affected with aging time than maximum stress. It can be concluded that increasing aging time has an accelerating effect on the changes of mechanical properties of the elastomeric structure.

From the DSC results, acetyl ferrocene used as burning rate catalyst in the HTPB-based elastomeric system, first degradation temperature drastically decreases from temperature level of 364°C to 315°C. This behavior can be attributed to a catalytic effect of acetyl ferrocene on the degradation temperature of the system.

Increasing crosslinking density in the barrier structure by introducing trifunctional aziridine brings about the inhibition of acetyl ferrocene migration in the HTPB-based elastomeric matrix.

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