# Bonding a HTPB Liner to Modern Rocket Case Materials

ROLAND SANDÉN and NIKLAS WINGBORG,* *National Defence Research Institute, Box 551, S-14725 Tumba, Sweden*

## Synopsis

Methods for bonding propellants based on HTPB (hydroxyl-terminated polybutadiene) to modern rocket motor case materials have been studied. Thus a typical rocket propellant liner, based on HTPB, was adhesion tested to various polymer materials and to an aluminum alloy. The polymer materials are an epoxy resin and composite materials, based on epoxy-poly(ether sulfone), poly(ether ether ketone), poly(amide imide), and poly(phenylene sulfide). With proper surface treatment, excellent results were obtained at separation tests for all materials with cohesive failure in the liner.

## INTRODUCTION

In a typical composite propellant rocket motor, the propellant is bonded to the motor case with an elastomeric material, known as a liner. This liner acts both as an adhesive and as a heat-insulating material. For the function of the motor it is of vital importance that the liner is bonded both to the propellant and to the motor case in a reliable way.

In motor constructions, steel alloys have so far been the materials most often used, but in order to save weight, new materials such as high-performance aluminum alloys and polymer composites are now creating much interest. With new materials, however, there is always a risk of adhesion problems. Both polymers and aluminum can be difficult to bond in a durable and reliable way. Before new materials are introduced in a construction, experimental tests should be carried out to secure good adhesion. A study of the surface and adhesion properties of poly(ether ether ketone) has been reported by Whitaker et al.,[1] and several methods for pretreating aluminum to enhance its adhesion properties have been suggested, for example, etching with acids, phosphating, and anodizing.[2] These methods are, however, complicated and time consuming. In this work experiments have been carried out with a typical HTPB-based liner, which has excellent adhesion properties to steel and to HTPB-based propellants. The intention of the work has been to find methods to obtain good bonding between this liner and modern motor case materials.

## EXPERIMENTAL

### Tested Materials

#### *Case Materials*

**Aluminum.** A high-performance alloy from Raufoss, Norway, "T6," containing Zn, Mg, Cu, and Co.

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**Epoxy.** From Ciba-Geigy, Switzerland. Based on Araldite LY556, curing agent HY917 and accelerator DY070. Mixed in proportions 100/90/1, respectively.

**Fibredux 914.** A glass fiber-laminated poly(ether sulfone)-modified epoxy material from Ciba-Geigy.

**PEEK.** Poly(ether ether ketone). A carbon fiber-laminated prepreg, APC-2, from ICI, England.

**PAI.** Poly(amide imide). Torlon 7130 from Amoco Chem. Corp., USA. This is the most metal-like of the different Torlon qualities. It contains 30% carbon fibers and 1% PTFE.

**PPS.** Poly(phenylene sulfide). Ryton R10 from Phillips Chemical Co., USA. Filled with glass fibers and mineral fillers.

### *Liner Components*

**HTPB.** Hydroxyl-terminated polybutadiene, R-45HT from ARCO, USA.

**BKF.** 2,2′-methyl bis(4 methyl-6-*tert*-butyl phenol) from Bayer AG, West Germany.

**DDI.** Diisocyanate 1410 from General Mills Chem. Inc, USA. Curing agent for HTPB.

**Carbon Black.** Sevacarb MT from Seven Valley Chemical Industries Ltd, USA.

**Aerosil 220.** Synthetic silicate from Degussa AG, West Germany. A fluffy material used as a thixotropic agent.

The composition of the liner was as follows:

| Component | % |
|---|---|
| HTPB | 61.0 |
| BKF | 1.2 |
| DDI | 17.7 |
| Carbon black | 18.3 |
| Aerosil | 1.8 |

### *Primers*

**Desmodur R.** A 20% solution of triphenylmethane triisocyanate in dichloromethane, from Bayer AG, West Germany.

**Desmodur RF.** A 20% solution of thiophosphoric acid tris(*p*-isocyanatophenyl) ester in dichloromethane, from Bayer AG, West Germany.

## Test Method

### *Peel Test*

What is called a peel test close to international standard ISO 813–1986, was used. The specimens were produced from the different case materials. They were first washed with trichloroethylene and acetone and then, in some cases, further surface-treated (e.g., ground with a fine sandpaper, blasted, or primed). The liner mixture was applied, reinforced with a glass fiber web, and cured (7 days at 70°C). The tensile experiments were carried out within 6 days after

TABLE I
Adhesion of HTPB Liner to Aluminum (Peel Test)

| Surface treatment of the metal | Adhesion value (N/mm) | Cohesivity (%) |
|---|---|---|
| Blasting | 0.42 ± 0.12 | 0 ± 0 |
| Blasting + Desmodur RF | 0.17 ± 0.02 | 0 ± 0 |
| Blasting + Desmodur R | 1.42 ± 0.09 | 100 ± 0 |

Six specimens were used in each case.

curing. The adhesion value is given by the force needed to separate a strip of the cured liner from the test material at an angle of 90°. The strain rate was 10 mm/min and the temperature was kept within 23 ± 2°C for 16 h before and during the test. It was also observed whether the fracture was of adhesive or cohesive type. In cases when the fracture was partly cohesive the percentage of the surface of the case material covered with the liner material was estimated. This percentage is called the cohesivity.

## RESULTS AND DISCUSSION

Results from the peel tests are given in Tables I–III. The effect of different pretreatments of the aluminum surface is shown in Table I. As can be seen, treatment by washing and blasting alone is not enough to give a good adhesion. Priming is also necessary and the choice of primer is very important. Desmodur R gives a very strong adhesion, while Desmodur RF seems to cause a decrease in the adhesivity.

To determine if the aging of the oxide surface of aluminum has any influence on the adhesion, experiments with various times, $t$, between blasting and primer treatment were carried out. The results are given in Table II. In all cases, the adhesion was so good that cohesive failure occurred in the liner material and no change with aging time could be seen.

In Table III are shown the results from adhesion experiments with the polymer materials. As can be seen, simple surface-treating methods were enough to obtain a strong bonding. With the PEEK, PAI, and PPS materials light grinding and treatment with Desmodur R was enough, With Fibredux 914 light grinding was enough and with the epoxy material the liner could be cured directly against the surface without any special pretreatment.

TABLE II
Adhesion of HTPB Liner to Aluminum

| $t$[a] (h) | Adhesion value (N/mm) | | Cohesivity (%) | |
|---|---|---|---|---|
| 0 | 1.20 | 1.26 | 100 | 100 |
| 0.5 | 1.52 | 1.33 | 100 | 100 |
| 1.0 | 1.56 | 1.34 | 100 | 100 |
| 170 | 1.48 | 1.24 | 100 | 100 |

[a] Time from blasting to treatment with primer Desmodur R. Peel test.

TABLE III
Adhesion of HTPB Liner to Different Polymer Materials: Effect of Surface Treatment[a]

| | Epoxy | | | Fibredux 914 | | | PEEK | | | PAI | | | PPS | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Surface treatment | n | a v (N/mm) | C (%) | n | a v (N/mm) | C (%) | n | a v (N/mm) | C (%) | n | a v (N/mm) | C (%) | n | a v (N/mm) | C (%) |
| — | 6 | 1.26 ± 0.06 | 100 ± 0 | 2 | 0.13 ± 0.05 | 0 ± 0 | 2 | 0.10 ± 0.02 | 0 ± 0 | 2 | 0.6 ± 0.1 | 0–5 | 2 | 0.16 ± 0.06 | 0 ± 0 |
| Grinding | | | | 4 | 1.1 ± 0.2 | 100 ± 0 | 2 | 1.04 ± 0.02 | 0–85 | 6 | 1.3 ± 0.6 | 10–90 | 2 | 0.18 ± 0.03 | 0 ± 0 |
| Primer | | | | | | | 3 | 1.0 ± 0.1 | 40–60 | 4 | 1.1 ± 0.2 | 100 ± 0 | 6 | 1.2 ± 0.6 | 40–90 |
| Grinding + primer | | | | | | | 6 | 1.1 ± 0.1 | 100 ± 0 | 6 | 1.4 ± 0.3 | 100 ± 0 | 6 | 1.23 ± 0.06 | 100 ± 0 |

[a]Abbreviations: C = cohesivity; n = number of specimens in each case; a v = adhesion value. Primer: Desmodur R. Peel test.

The results show that very good bonding can be obtained. To measure the strength of the adhesion in a proper way, adhesive failure is needed. In view of this, no proper adhesion values have been obtained in the cases when strong bonding occurred. Concerning the purpose of obtaining good bonding of the liner to the case materials the results are, however, very clear. Either the adhesion was excellent and too strong to measure or it was too weak to be of any interest. In a specific case, complementary tests should be carried out with a proper motor which should be stored at different temperatures and also test fired to secure no breakage at the long-duration stresses and strains that will occur at such tests.

The choice of primer is extremely important. The two primers tested are both of the isocyanate type but they may give quite different results. For the aluminum alloy with Desmodur RF, no increase in the bond strength was obtained, while Desmodur R has proven to be very effective.

Some specimens from all the case materials were stored at room temperature for 6 months and then peel-tested. Essentially the same results were obtained and no deterioration with time could be seen in any case.

## CONCLUSIONS

The results show that strong adhesion of the liner to the case materials can be obtained with simple methods such as light grinding and priming. The primer Desmodur R is in these cases very efficient.

## References

1. R. Whitaker et al., Characterization and Adhesive Bonding of Poly(ether Ether Ketone) Resins and Composites, MLM-3164 (OP) DE 85-002526, 1984.

2. D. M. Brewis, Ed., *Surface Analysis and Pretreatment of Plastics and Metals*, Applied Science Publishers, London, 1982.

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