

Case Bonded System for Composite Solid Propellants

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The examination results of eleven different liners, based on hydroxyterminated polybutadiene (HTPB), carboxyterminated polybutadiene with and without acrylonitrile (CTPB, CTBN) and seven solid rocket propellant types, also based on hydroxyterminated polybutadiene and on polyurethane polymer (PU), are presented in this paper. Viscosity values, mechanical characteristics (maximum stress and strain at maximum load) and adhesive strength between propellant/liner and metal/liner were determined.

Key words: composite rocket propellants, polybutadiene, polyurethane, liner, adhesion, maximum stress, strain at maximum load.

Introduction

IN a rocket motor with a built-in solid rocket propellant grain, the composite propellant (CRP) is bonded to the motor case with an elastic material, known as a liner. This liner acts both as an adhesive (called: liner) and as a heat-insulating material (insulation), (in the further text: liner). For the functioning 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 [1]. The performance and reproducibility of the desired characteristics of a solid propellant grain are highly dependent upon the adequacy of the liner in achieving the bonding of the grain to the case in order to establish stable-state burning and realisation of ballistic requirements. Upon burning, the grain produces a large volume of a highly-pressurized gas exhausted from the chamber through the nozzle at high velocity. The reaction resulting from the acceleration of gases through the nozzle creates the propulsive thrust [2]. The motor casing insulation is also required to protect the casing from the eroding effects of hot burning gases [3].

The liner placed between the case and the propellant serves as a structural material to transmit stress to the load-bearing components. The bond that the liner forms between the propellant and the chamber walls prevents movement of the propellant grain, which would create air voids, spaces and uncontrollable burning due to the change of pressure value, failure appearance, motor accident and explosion [2, 4]. The propellant grain is securely held in the motor case throughout motor firing, although nozzle blockage, unpredictable variations in burning surface, over-pressure and even case rupture can occur. At these extreme situations, the liner polymer binder, owing to its mechanical characteristics, has to provide an adequate strain value to compensate for the thermal stress occurring during temperature change due to different coefficients of motor case and grain dilatation; necessary strength to resist to detrimental effects of shocks and frictions during handling; desired waterproof quality and insulation during burning.

The major problem in obtaining good adhesion between

the composite propellant and the liner concerns the bonding of the dissimilar materials used in the propellant grain and the liner [4]. The liner composition has to be compatible with propellant components and to simultaneously bond well with them. It is very often a case that the binder of the liner has been based on the same polymer as the propellant for the grain. The diffusion process has to be considered in the liner selection [5]. In some types of propellants which have shown very good bonding, the plasticizer migration after the propellant grain is cast caused weakening of the otherwise strong bond. The failure was described as cohesive within the propellant, meaning that the propellant became weaker, not the adhesive bond. The IR spectrophotometry examination of even blank surfaces after separation by the peel test showed the proof of cohesion peeling. The location of failure (near the interface) and the mode of failure (cohesive within the propellant) suggest that the propellant cohesive strength is the limiting factor in the bond strength of this type of systems. All the components which soften the propellant make the bond weaker: higher content of plasticizer, acids, amines and other components which contain active hydrogen and moisture necessary for the curing agent to be cured in the liner or the propellant. The stability of the propellant/liner bond, and thereby of propellant itself, can be examined by the method of accelerated aging, i.e. by storing the samples at elevated temperatures and determining the values in real conditions by data interpolating.

The choice of a proper filler is most important for a liner as an insulation agent. The liner adopts thermal conductivity and insulation density, where these two values are approximately the same for the binders. Fillers provide structural integrity and resistance at high temperatures, and protect integrity from the influence of gas flows. Other additives can be represented up to 50 % of the total mass. [6]. When the liner acts as an insulator which inhibits the grain surface and thereby prevents the burning of the external wall, the different fillers are used:

– Coolants – oxalates, carbonates, ammonium compounds;

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- Flame retardant fillers – phosphates, chalcogenates;
- Refractory fillers – zirconium oxide, titanium dioxide, alumina oxide, silica;
- Melt of char forming fillers – boron oxide, silica, phenol resins;
- Low density fillers – phenol resins (hardablatives)
- Glow suppressants- antimonyoxide
- Hydrated fillers for transpirational cooling – boric acid

The most commercial elastomers include silica (non hygroscopic) and asbestos as fillers. A very effective method for preventing moisture bonding, which migrates the bonds inside and degrades them, is drying. Many liners are designed to provide good reological properties of uncured liners (viscosity, gelling characteristics and pot life). The requirements must be in accordance with proposed parameters and limitations.

A liner composition has to satisfy grain production technology requirements. Preparing the surface for liner coating is a very important operation: it has to be rough, clean and dry [5]. The liner/propellant bonding is formed by curing the propellant in contact with the cured liner.

The propellant/liner bond is one of the most difficult areas of a solid rocket motor to describe quantitatively. The characterization of this surface is only analyzed as an independent part in the rocket motor development procedure.

Experimental part

For the purpose of determining liners which satisfy the established requirements, eleven different compositions have been made.

The liners consisted of two components:

- Polymer (resin) - carboxyterminated copolymer of butadiene and acrylonitrile, (type CTBN 1300×15), hydroxyterminated polybutadiene, (HTPB, two types R45-HT and HB70) and carboxyterminated polybutadiene, (CTPB, type Butarez CTL II).
- Curing agents: - tris-1-(2-methylaziridynyl) phosphin oxide (MAPO), epoxide curing agents (EPON 812), isophorone-diisocyanate (IPDI) and toluene-diisocyanate (TDI).

Polymers with carboxyl groups, due to intermolecular association, have higher viscosity values than HTPB polymers (30 Pas/25°C). EPON 812 (glycerol triglycidyl ether) and MAPO are curing agents for carboxyl polymers [7]. The liners with CTBN polymers are used in the case of demand for liners with a good low-temperature mechanical characteristics, which are due to secondary nitrogen atom bonds in the chain structure, (though the viscosity values are higher (70 Pas). HTPB polymers have good values at low-temperature strains and high-temperature stresses. Their viscosity values are at the lowest (4 Pas - 7.5 Pas).

The hardness and thermal resistance were obtained by adding fillers, such as carbon black (CB), talck (T), cement (C) and asbestos (A).

The different additives were added into this mixture in order to improve oxidation processes and aging resistance or to provide the necessary time for the motor case casting [5], such as dioctyladipate (DOA), triethylenetetramine (TET), phenyl-β-naphtylamin (FβNA), 2,2'-methylenebis [6-(1,1-dimethylethyl)-4-methylphenol] (AO22).

Based on the former investigations [5], [7], the chosen liner compositions were homogenized in the 4PU mixer and shown in Tables 1, 2 and 3. The curing time was 48 hours at (70±2)°C [8].

Table 1. Liners based on CTPB prepolymers (%)

Batch	CTL II	Carbon black	C	A	MAPO	EPON
1	72.1		14	10	2.9	
2	70.7		15	10		3.6
7	87.7	7.0 ⁴ +1.8 ⁵			3.5	
9	87.1	4.4 ⁴ +1.1 ⁵			3.5	
10	81.7	5.0 ⁴		10	3.3	

Table 2. Liners based on HTPB prepolymers (%)

Batch	HTPB	Curing	Carbon Black	T	C	DOA
4	69.3 ¹	4.6 ⁶		10	15	
3	67.2 ¹	4.6 ⁶	26.6 ³			1.0
8	69.3 ²	4.6 ⁶		10	15	
11	66.2 ²	5.8 ⁷	14.8 ⁴ +4.9 ⁵			7.9

Table 3. Liners based on CTBN prepolymers (%)

Batch	CTBN	EPON	CB	C	A	TET
5	68.8	5.7		14.9	9.9	0.1
6	73.3	6.0	20.0 ³			

¹ – HTPB, Sartomer, ² – HTPB, HB70

³ – SRC CB, ⁴ – 50%, basic, ⁵ – 100% tip I

⁶ – TDI, ⁷ – IPDI

Viscosity of uncured liners was determined at the Brookfield model [7]. The liner samples were obtained by casting on Teflon pans, and after curing specimens for uniaxial mechanical testing were cut [9]. Maximum stress, (σ_m) and strain at maximum load (ϵ_m) were determined.

The composite rocket propellants, as samples for liner foundation, are produced with the binder (B) based on polyoxipropylene dyol/tryol mixture, called polyurethane (PU) and based on HTPB. The already mentioned curing agents (CA) IPDI and TDI were used as well as dimeryldiisocyanate (DDI) [10]. Ammoniumperchlorate (AP) was used as an oxidizer at three different fractions – average particle sizes (200 μm, 80 μm and 10 μm), while aluminium (Al) and carbon black were burning stabilizers. The mixing was performed at 50±2°C, and the propellant was cured for 120 hours (HTPB) or 72 hours (PU) at 70±2°C. The specimens were made from the block sample for uniaxial tensile tests of mechanical characteristics tests at 20±3°C on the Instron tester type 1122 [11], [12].

The specimens for the determination of adhesive characteristics were obtained from the samples of propellants and liners. [13]. Propellant compositions (CRP) are shown in Table 4.

Table 4. Composition of CRP

CRP batch	B/CA	AP mixture	AP (%)	additives (%)	Solid phase, (%)
H1	HTPB/IPDI	200/80	79.0	čad 0.5	79.5
H2	HTPB/TDI	200/10	76.5	Al 4.5	81.0
H3	HTPB/DDI	200/10	76.5	Al 4.5	81.0
H11	HTPB/IPDI	200/10	76.5	Al 4.5	81.0
H12	HTPB/IPDI	200/10	81.5	Al 4.5	86.0
PU1	PU/TDI	200/10	76.0	-	76.0
PU2	PU/TDI	200/10	82.0	-	82.0

The specimens for testing the metal/liner adhesive characteristics were obtained in two ways:

1. Liners were applied on the cylindrical case by rotation, cured, then one side of the case was cut into motor case/liner strips and finally their bonding values were determined.
2. Liners were cast into a 4mm thick teflon pan, then steel quadrants were immersed into the liner up to a certain depth (controlled by auxillary tools) to provide a contact with the uncured layer of the liner.

Results and discussion

The viscosity values of the liner compositions can be seen in Table 5.

Table 5. Liner viscosity values

Label of liner	Polymer type	VISCOSITY (Pas) during the time (min)			
		15	30	45	60
1	CTPB	12.8	12.2	12.2	12.8
2	CTPB	18.6	19.8	-	21.8
3	HTPB	34.5	53.8	67.2	74.2
4	HTPB ¹	22.4	31.4	-	44.2
5	CTBN	32.0	35.2	-	44.8
6	CTBN	24.3	32.0	35.2	-
7	CTPB	HARDLY CASTABLE			
8	HTPB ²	9.6	13.4	16	-
9	CTPB	92.2	98.6	103.0	-
10	CTPB	153.6	159.4	167.0	-
11	HTPB	HARDLY CASTABLE			

The shown values imply that:

- Use of carbon black increases viscosity, particularly if it is fine and surface active (11).
- the change of fillers from carbon black (6) to cement and asbestos (5) in the composition with CTBN does not considerably effect viscosity,
- in case of CTPB, aziridynyl systems (MAPO) gave lower viscosity than epoxide systems (EPON) (1 and 2). Higher increase in viscosity was obtained by applying carbon black, basic type, [14], while the filler amount which system can accept was smaller (7, 9 and 10). Liner 10 has the highest level of measured viscosity values, but also the highest quantity of the solid phase responsible for strength [15].

Testing of liner mechanical characteristics was carried out immediately before the propellant casting over them [7]. The examination results are presented in Table 6.

Table 6. Mechanical characteristics of liners

Label of liner	σ_m (daN/cm ²)	ϵ_m (%)	E (daN/cm ²)
1	4.80	104.30	7.12
2	5.99	94.36	9.48
3	11.49	90.86	17.52
4	6.14	185.16	6.38
5	7.99	47.96	17.90
6	12.39	405.38	6.78
7	12.92	168.60	9.33
8	7.80	259.78	8.81
9	8.49	197.85	4.96
10	11.56	160.64	9.09
11	40.90	552.26	9.67

Compositions 3, 6, 7, 10 and 11 from Table 6, have maximum stress values higher than those for the propellants (>10 daN/cm²), which points out their good characteristics. The other compositions have shown satisfactory values (5, 8 and 9) as well, which is in accordance with the literature - 12 daN/cm² (σ_m) and min 270 % (ϵ_m) after 1.5 h curing time at 120°C [15]. The effect of TET is visible in compositions 1 and 2 with small surface activity fillers.

Liners 4 and 8 are the same composition, but with different types of HTPB prepolymers, where the HB70 has proved to be a better matrix (and the viscosity as well, Table 5). The values of hardly cast composition 11 are over the expected ones, and due to the HB70 prepolymer and the

IPDI curing agent, it is possible to homogenize more reactive types of carbon black (basic and type I) than the SRC carbon black [10].

The strains at maximum load in liners No 6, 7, 8, 9, 10 and 11 are in the range from 160 % to 400 %.

The propellant mechanical characteristics are given in Table 7.

Table 7: Mechanical characteristics of CRP

Batch	σ_m (daN/cm ²)	ϵ_m (%)
H1	6.95	12.24
H2	7.73	21.08
H3	7.54	14.29
H11	6.60	36.07
H12	8.65	20.50
PU1	2.31	138.48
PU2	4.09	48.73

The measurements of the adhesive characteristics of the propellant/liner specimens can be seen in Table 8.

Table 8. Bond strength of propellant/liner specimens

No	Longitudinal load (N/cm)							
	liner	H1	H2	H3	H11	H12	PU1	PU2
1		8.6	3.9	3.2	2.9	2.4	7.6	6.0
2		5.4	4.5	3.9	2.9	1.5	7.6	9.0
3		3.7	3.2	2.1	2.9	2.3	9.8	9.2
4		7.2	4.9	5.2	3.8	3.4	8.6	6.4
5		3.7	2.6	1.9	1.0	0.6	9.5	7.6
6		5.4	5.0	4.5	2.6	2.1	12.5	9.2
7		3.6	3.6	2.4	3.2	1.6	9.0	4.6
8		11.3	6.4	5.6	8.0	10.7	15.9	14.8
9		3.1	3.4	2.6	2.2	1.8	6.4	3.3
10		3.1	3.2	3.2	1.8	1.6	7.9	4.4
11		4.9	7.0	5.8	4.4	5.8	8.2	7.2
Mean value		5.5	4.3	3.7	3.2	3.1	9.4	7.4
Sy		2.6	1.4	1.4	1.9	2.9	2.7	3.2

The PU propellants have shown the highest adhesive values (PU1 and PU2), and those with lower solid phase content (PU1) have shown maximum values and the smallest deviations depending on the liner composition.

The adding of the fine AP fraction decreased the bond strength values in regard to the values for the propellant batch H1 (mixture of 200/80 AP) and the batch H11 (mixture of 200/10 AP) [16].

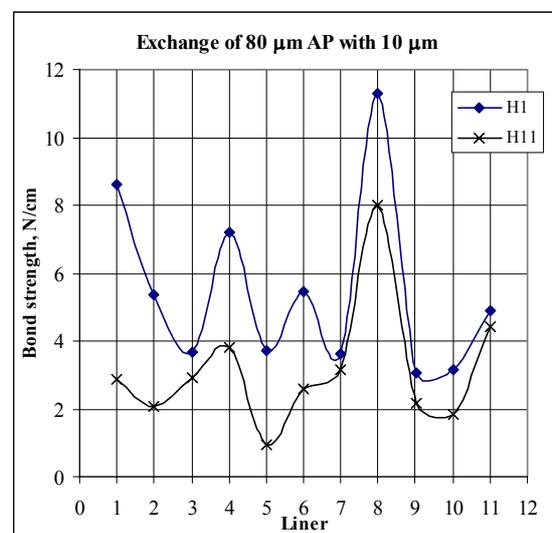


Figure 1. The effect of changing a fine AP fraction from 80 µm to 10 µm on the bond strength

A larger degradation of bond strength was recognized in the case of the propellant (with the HTPB polymer) and the CTPB- or CTBN -based liner (1, 2, 5, 6 and 10), but all types of fillers, except carbon black (basic and type I), have shown a greater reduction of the bond strength, as it can be seen in Fig. 1.

The highest values of the bond strength were attained in the case of TDI as a curing agent in the binder of the propellants with the same AP fraction (H2, H3 and H11), by changing the curing agents in the binder, except in the case of liner 8 which consists of HB70 - the polymer used in all HTPB polymers in this examination. The change of liner compositions is given in Fig. 2.

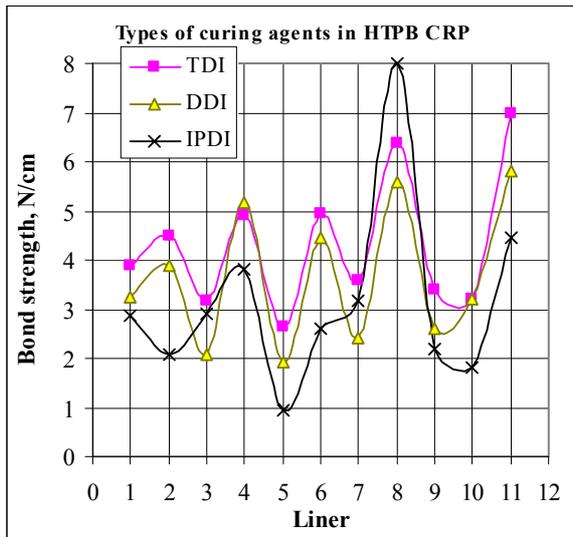


Figure 2. The effect of the propellant curing agent changing on the bond strength

The increase of the binder content in the propellant ground has positive effects on adhesive characteristics, as expected in regard to literature [17]: the values of the liner at the propellant substrum with 16% of the binder were from 3.85 N/cm to 4.9 N/cm, but at the propellant substrum with 12% of the binder the values were only 1.6 N/cm. The effect of the propellant binder content for two binder types and the solid phase propellant contents given at two levels (HTPB: 81 mas.% - H11 and 86 mas.% - H12 and PU: 76 mas.% - PU1 and 82 mas.% - PU2) can be seen in Fig. 3.

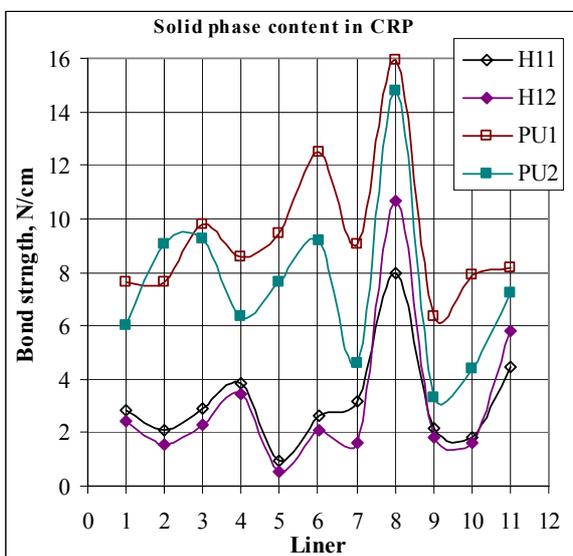


Figure 3. The effect of the propellant binder content for two propellant types

The change of the maximum stress and the bond strength for all liner compositions has been shown in Fig. 4. There is a phase shift of the respective extreme curve points, i.e. the liners with the highest maximum stress values have the lowest adhesive properties (example: H1 propellant).

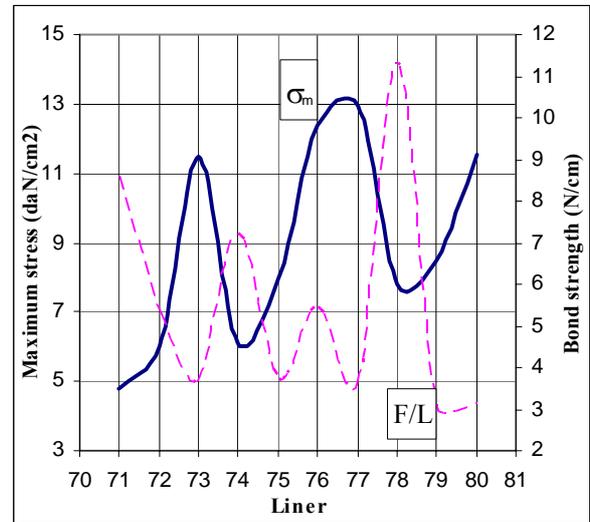


Figure 4. The change of the maximum stress and the bond strength for one propellant composition

The change of the strain at maximum load and the propellant/liner bond strength for all liner compositions has been shown in Fig. 5. The values of adhesive characteristics are proportional to the strain values and the trends of the curves are identical. The maximum bond strength values (higher than 5-5.5 N/cm) are in the strain region from about 200% - 260%, but with the further strain increase the adhesion dropped. The final effects depend on the liner components, but carbon black has proved to be a universal filler for all systems.

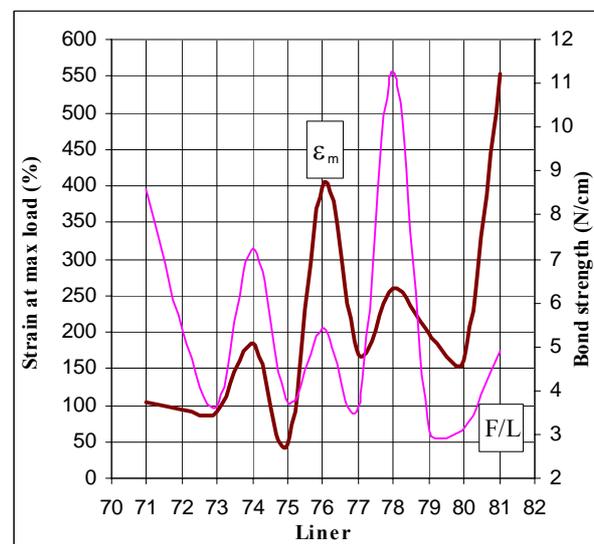


Figure 5. The change of the strain at maximum load and the bond strength for one propellant composition

The metal/liner bond strength investigations were performed on the strips cut from the cylindrical case coated on the interior side with a 15 μm thick alkid melamine layer of lacquer and three types of liners over it. Liners No 3, 8 and 10 were applied on these case strips. The deformation rate was 50 mm/min [5]. The bond strength was higher than the

strength of the liner, because of the liner rupture, so the load values presented the measure of the liner tensile properties, while the adhesive properties were considerably higher. The results of these examinations are given in Table 9.

Table 9: Metal/liner bond strength (motor case)

Label of liner	T (°C)	Sample number	Load F, (N)	Longitudinal load F/l, (N/cm)
8	20	1	-	-
	50	2	10	4.0
	-50	3	67	26.3
10	20	1	38	15.5
	50	2	22	9.1
	-50	3	25	10.4
3	20	1	54	22.2
	50	2	25	9.8
	-50	4	114	47.5

The metal/liner bond strength was examined on the samples without the melamine layer [5]. The results of these examinations are given in Table 10.

Table 10: Metal/liner bond strength (quadrant)

Label of liner	Curing time, h	Load F, (N)	Longitudinal load F/l, (N/cm)
1	24	14	4.52
	90	13	4.71
2	24	12.5	4.44
6	24	25	8.3

The peeling of the liner from the metal was manifested only on sample 6, before the rupture of the liner and these values are significantly higher than those obtained on liner samples 1 and 2. This is understandable when the mechanical characteristics of these two compositions are compared: for 1 and 2 (~5 and ~6 daN/cm²) in relation to No 6 (~12 daN/cm²). The given results show that the liner mechanical characteristics represent the limiting factor more than the adhesive characteristics of the metal/liner bond.

The appearance of examined samples can be seen in Fig.6.



Figure 6. Separation of the liners from the metal samples

Conclusion

The examinations represented in this paper have been related to different liner compositions based on three types of polymers: carboxyterminated polybutadiene, hydroxyterminated polybutadiene and carboxyterminated copolymer polybutadiene acrylonitrile. Seven composite rocket propellant types have been prepared as substrum based on hydroxyterminated polybutadiene and on polyurethane polymer.

In the case of CTPB, the aziridynyl systems have shown lower viscosity than the epoxide systems. The viscosity of CTBN compositions has satisfied the values which have not changed significantly by filler varieties, but it is still higher than those of the CTPB polymers. The lower viscosity values were obtained by the use of the HB70 type of the HTPB polymer other than R45HT type in liner compositions. Higher increase in viscosity was achieved by applying carbon black, basic type, and as a consequence, a smaller filler amount was required.

Among 11 liner compositions, only three showed the values lower than 7 daN/cm², but five of them had higher values than 11 daN/cm². These levels of maximum stress, the same as those of the propellants, could be achieved by using all three types of polymer matrices and with carbon black as a filler. Also, out of all these liners, three of them have strains at maximum load lower than 100 %.

In concluding this paper, it can be seen that:

- The liners applied on PU propellants have shown the highest adhesive values, and those with a lower solid phase content have shown the maximum values and the smallest deviations depending on the liner composition,
- the adding of the fine AP fraction decreased the bond strength values, especially when the polymer matrix of the liner and the propellant are different, where the decreasing effect has been seen in any filler present, except carbon black (basic and type I),
- the highest values of the bond strength were attained in the case of TDI as a curing agent in the binder of the propellants, except when a liner contained the HB70, the polymer used at all HTPB propellant polymers,
- increasing the binder content in the propellant ground had a positive effect on the adhesive characteristics of all the liners,
- the minimum values of the curve connecting the bond strengths for all the liners overlap with the maximum values of the curve showing the change of maximum stresses of the same liners, i.e. the liners with the highest maximum stress values have the lowest adhesive properties,
- values of the adhesive properties are proportional to strain values, i.e. the trends of the curves are identical and are not shifted as in the case of the maximum stress. If the curing temperature is higher, the strain at the maximum load will be lower, as well as the propellant/liner bond strength,
- maximum bonding values (higher than 5-5,5 N/cm) are in the strain region from about 200 % - 260 %, but with the further increase of strain, the adhesion drops.

The experimental values presented in this paper are in accordance with literature, which means that the usage of the mentioned raw materials results in liners of satisfactory characteristics.

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Lajneri za kompozitna raketna goriva

Prikazano je istraživanje jedanaest sastava lajnera na bazi karboksiterminiranog i hidroksiterminiranog polibutadiena i karboksiterminiranog kopolimera butadiena i akrilonitrila. Izrađeno je i sedam sastava kompozitnog raketnog goriva na bazi hidroksiterminiranih polibutadiena i poliuretana. Određivane su vrednosti viskoziteta, zatezne čvrstoće i izduženja pri maksimalnoj sili svakog izrađenog lajnera, kao i vrednosti adhezionih karakteristika veze gorivo/lajner i metal/lajner.

Ključne reči: kompozitna raketnagoriva, polibutadien, poliuretan, lajner, adhezija, mehaničke karakteristike.

Liniers pour les propergols composites

Dans ce papier on a présenté les résultats de l'examen de 11 compositions de liniers, à la base de polybutadiène carboxyterminé et hydroxyterminé, copolymère polybutadiène carboxyterminé et acrylonitrile. Sept types de propergols solides ont été réalisés à la base de polybutadiène hydroxyterminé et polyuréthane. On a déterminé les valeurs de la viscosité, la résistance à la tension et l'allongement à la force maximale de chaque liner élaboré ainsi que les valeurs des propriétés adhésives chez les couples propergol /liner et métal/liner.

Mots clés: propergols composites, polybutadiène, polyuréthane, liner, adhésion, caractéristiques mécaniques.