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OPTIMIZATION OF INTERFACIAL BONDING OF EPDM-BASED THERMAL INSULATION AND STEEL ROCKET MOTOR CASE-DEVELOPMENT OF THE SYSTEM OF ADHESIVE LAYER AND CEMENT

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Abstract: *The design of rocket motors with case-bonded propellant grains requires reliable thermal protection systems capable of protecting the motor case from high thermal and mechanical loads during operation. Due to that, it is important to apply thermally stable insulating material using a suitable technology and achieve good adhesion between thermal insulation and rocket motor case. This paper presents the development of the system of adhesive layer and cement for optimal interfacial bonding of EPDM based thermal insulation and steel rocket motor case. An EPDM thermal insulation compound reinforced with chopped and milled carbon fibers was selected for application to the rocket motor case and further evaluated through thermal analysis and adhesion testing. The gluing of EPDM-based thermal insulation to the interior of the rocket motor case was performed by vulcanization in an autoclave using the adhesive layer and cement as adhesion promoters. Thermal stability of the insulation material was evaluated by simultaneous thermal analysis (STA), while adhesion between rubber-rubber and rubber-steel was determined according to ASTM D1876 and ASTM D6862 standards. The results confirmed satisfactory adhesion properties of the selected adhesive system and adequate thermal stability of the insulation compound. The rocket motor case was successfully lined with the developed EPDM-based thermal insulation in a thickness range of 1.8–2.2 mm and vulcanized under industrial processing conditions.*

Keywords: *EPDM rubber, thermal insulation rocket motor case, adhesion, thermal protection*

1. INTRODUCTION

Rocket motors with case-bonded propellant grains require thermal protection systems capable of protecting the motor case from the extreme thermal and mechanical loads generated during motor operation . Thermal insulation is applied directly to the inner wall of the motor case to reduce heat transfer from combustion gases and prevent overheating of the metallic structure. In addition to thermal protection, the insulation layer may also serve as an inhibitor, protecting selected propellant surfaces during combustion [1, 2, 3].

Elastomer-based thermal insulation materials are widely used in rocket motor applications due to their processability, elasticity and thermal stability. Among them, EPDM rubber is of particular interest because of its resistance to thermal ageing and oxidation. In the investigated thermal insulation compound, chopped and milled carbon fibers, ammonium polyphosphate and melamine were used to improve thermal protection performance [4, 5, 6].

A critical requirement for the reliable operation of case-bonded rocket motor systems is the interfacial adhesion between the thermal insulation material and the metal motor case. Insufficient adhesion may lead to delamination, local heat accumulation and degradation of structural integrity during service [7]. For this reason, the development of the system of adhesive layer and cement and the suitable vulcanization technology must ensure an optimal interfacial bonding between thermal insulation and the metal rocket motor case.

The aim of this work was to optimize the interfacial bonding of EPDM-based thermal insulation through the development of a system of adhesive layer and cement and the development of suitable vulcanization technology for applying an EPDM-based thermal insulation to the inner wall of a steel rocket motor case.

2. EXPERIMENTAL

An EPDM-based thermal was used for application to the inner wall of a steel rocket motor case. The thermal

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insulation was designated TG-H-727/3. The composition of the investigated thermal insulation compound is presented in Table 1. A sulfur-based curing system consisting of mercaptobenzothiazole (MBT), tetramethylthiuram disulfide (TMTD), and sulfur was used for vulcanization of the experimental compounds. Commercially available processing additives were also incorporated at optimum levels to improve processability. The thermal insulation was designated TG-H-727/3. Homogenization of the compounds was performed on a two-roll mill. After mixing, test specimens were prepared and curing conditions were determined from rheological measurements, while thermal stability was evaluated by STA.

Table 1. Formulation of EPDM-based thermal insulation (TI) compound

Components for TI compound formulation (TG-H-727/3)	Parts per hundred rubber / Mass percentage (phr/%)
EPDM rubber Dutral TER 9046	100.0 /42.9
Silicon dioxide, SiO2 VULKASIL S	15.00/6.4
Semi-reinforcing carbon black N550	15.0/6.4
Melamine	35.0 /15.0
Ammonium polyphosphate (APP)	35.0 /15.0
Carbon fibers, milled 0.1 mm	5.0/2.1
Carbon fibers, chopped 3.0 mm	5.0 /2.1
Chlorinated paraffin oil	3.5 /1.5
Paraffinic oil RPO320	10.0 /4.3
Zinc oxide, ZnO	5.0 /2.1
Stearic acid	1.0 /0.4
Sulfur	1.5 /0.6
MBT – Mercaptobenzothiazole	1.5 /0.6
TMTD – Tetramethylthiuram disulfide	0.5 /0.2
Total:	233.0 /100.0

An adhesive layer designated TG-HD-727/C3 and a cement designated TG-D-727/C6 were developed to provide bonding between the thermal insulation layer and the metal substrate. The compositions of the adhesive layer and cement are presented in Tables 2, 3 and 4, respectively. Adhesion properties were determined using peel and shear tests according to ASTM D1876 and ASTM D6862.

Table 2. Formulation of the adhesive layer compound

Components for adhesive layer formulation (TG-HD-727/C3)	Parts per hundred rubber (phr/%)
EPDM rubber Keltan 6950 C (or Dutral TER 9046)	60.0/28.2
Polychloroprene rubber, CR rubber, Baypren 110	40.0/18.8
Silicon dioxide, SiO2 VULKASIL S	15.0/7.1
Melamine	35.0/16.5
Ammonium polyphosphate (APP)	35.0/16.5
Zinc oxide, ZnO	5.0/2.4
Stearine	1.0/0.5
Magnesium oxide, MgO	1.0/0.5
Chlorinated paraffin oil	10.0/4.7
Adhesion promoter, Elastobond T 6000	2.0/0.9
Adhesion promoter, Elastobond T 6000	5.0/2.3
Sulfur	1.5/0.7
MBT–Mercaptobenzothiazole	1.5/0.7
TMTD–Tetramethylthiuram disulfide	0.5/0.2
Total:	212.5/100.0

Table 3. Composition of TG-D-727/C6 cement

Components	Parts per hundred rubber (phr/%)
CR rubber, Baypren 110	100.0/63.7
Silicon dioxide, SiO2 VULKASIL S	35.0/22.3
Plasticizer, Pacizer 6218	5.0/3.2
Zinc oxide, ZnO	12.0/7.6
Stearine	1.0/0.6
Accelerator, Rhenogran ETU-80	1.0/0.6
Magnesium oxide, MgO	1.0/0.6
Antioxidant / Anti-aging agent, Vulkanox SKF	2.0/1.3
Total:	157.0/100.0

Table 4. Composition of organic solvents and other components for TG-D-727/C6 rubber cement preparation

Components	Mass (g)
TG-D-727/C6	75.0
Petroleum ether / Gasoline	90.0
Toluene	90.0
Acetone	70.0
Resin, Koresin PLW	5.9
Adhesion promoter, Elastobond T 6000	4.6

The rocket motor case made of Č.4734 steel (according to the YUS) was prepared by abrasive blasting of the inner surface, followed by application of the system of adhesive layer and cement. The thermal insulation lining consisted of an adhesive layer with a thickness of 0.6 mm and a thermal insulation layer with a thickness from 1.4 mm to 1.6 mm, resulting in a total lining thickness between 1.8 and 2.2 mm.

After application, the lined rocket motor cases were vulcanized in an autoclave using direct steam at 150 °C and a pressure of 0.5 MPa for 40 min.

3. RESULTS AND DISCUSSION

3.1. Thermal Stability of the EPDM-based Thermal Insulation

The thermal stability of the investigated EPDM-based thermal insulation was evaluated by STA. The TG curve (Figure 1) showed a negligible mass loss below 250 °C, which can be attributed to the evaporation of residual low-molecular-weight components and moisture. The main degradation stage occurred between approximately 250 °C and 480 °C and corresponded to the thermal decomposition of the EPDM matrix and the release of volatile pyrolysis products. The compound exhibited a final mass loss of 71.87 %, while maintaining sufficient thermal stability throughout the temperature range relevant for the investigated application.

The DTG curve (Figure 2) revealed three characteristic degradation peaks located at approximately 298 °C, 384 °C and 464 °C. The first peak was associated with the decomposition of melamine and ammonium polyphosphate, the second with the formation of a protective carbonized layer, and the third with the primary degradation of the EPDM matrix. The obtained results confirmed the thermal behavior of the selected insulation compound and supported its use in the thermal protection system of the rocket motor case [8].

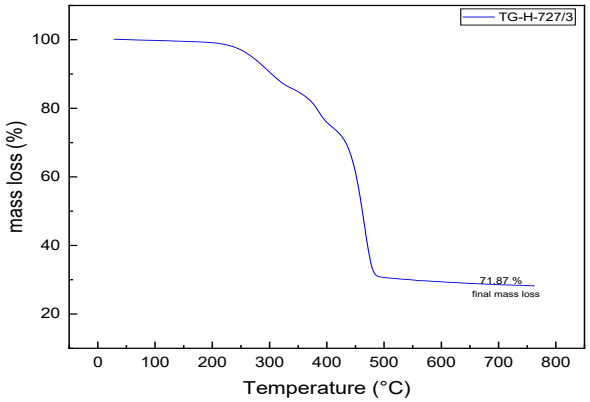


Figure 1. TG curve of the TG-H-727/3 formulations

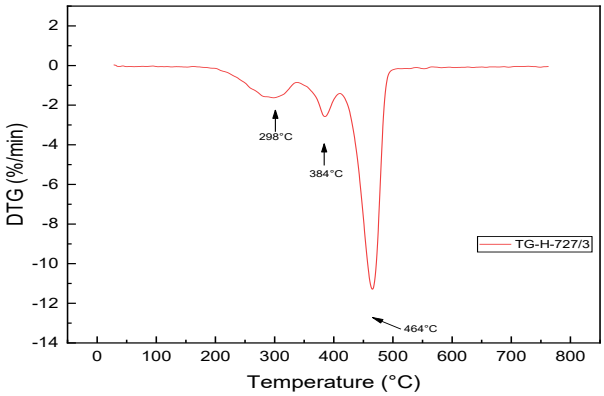


Figure 2. DTG curve of the TG-H-727/3 formulations

3.2. Adhesion Properties and Verification of the Application Technology

The sample codes used in the adhesion tests are defined as follows: T denotes the EPDM-based thermal insulation compound TG-H-727/3; P denotes the adhesive layer based on TG-HD-727/C3; C3 denotes TG-HD-727/C3 when used as a cement; C6 denotes the TG-D-727/C6 cement. For comparative rubber-rubber and rubber-metal adhesion testing, an adhesive based on natural rubber (NR), designated MC, was also used. The number in the sample code identifies the individual specimen [8].

The rubber-rubber peel test results (Table 5) revealed a strong influence of both the adhesive layer and the selected cement on the bond strength. The highest average peel strength (10.63 N/cm) was obtained for specimen T/P-C3-C3-T/P-C3, which contained the TG-HD-727/C3 adhesive layer and the C3 cement on both bonded surfaces. Failure occurred within the adhesive layer, indicating good compatibility between the thermal insulation, the adhesive layer and the cement system.
Specimens prepared without the additional adhesive-layer application exhibited significantly lower peel strength values. The average peel strength of specimen T-C3-T was 4.01 N/cm, while specimen T-C6-T showed the lowest value of 2.82 N/cm. These results demonstrate that the incorporation of the TG-HD-727/C3 adhesive layer considerably improves adhesion between thermal insulation layers. The intermediate value obtained for

specimen T/P-C6-C6-T/P-C6 (5.30 N/cm) suggests that the adhesive layer alone is not sufficient to achieve maximum bond strength and that the choice of cement also plays an important role.

Table 5. Rubber-rubber peel strength of the EPDM-based thermal insulation system

sample code	L (mm)	F_{max} (N)	F_{min} (N)	F_{avg} (N)	$F_{avg/L}$ (N/cm)	mean value (N/cm)
1-1 (T/P-C3-C6-T/P-C3)	31.65	27.00	24.00	25.32	8.0	6.70
1-2 (T/P-C3-C6-T/P-C3)	30.96	17.60	15.50	16.64	5.39	
2-1 (T-C6-T)	32.45	13.70	11.20	12.50	3.85	2.82
2-2 (T-C6-T)	31.28	7.10	4.00	5.63	1.80	
3-1 (T/P-C3-C3-T/P-C3)	30.83	27.88	26.60	27.70	9.04	10.63
3-2 (T/P-C3-C3-T/P-C3)	31.40	38.70	36.60	38.38	12.22	
4-1 (T-C3-T)	31.13	9.00	5.70	7.90	2.54	4.01
4-2 (T-C3-T)	32.04	18.70	16.80	17.56	5.48	
5-1 (T/P-C6-C6-T/P-C6)	31.67	21.00	14.50	16.79	5.30	5.30

The rubber–metal peel test results (Table 6) showed significant differences in adhesion performance among the investigated bonding systems. Specimens T-C6 (1-1), T-C6 (1-2), T-C3 (2-1) and T-C3 (2-2) represent the bonding systems based on C6 and C3, while T-MC represents the comparative MC adhesive. The highest peel strength was obtained for specimen T-MC, with an average value of 15.99 N/cm. Among the investigated C3 and C6 systems, specimen T-C6 (1-1) exhibited the highest peel strength of 5.03 N/cm. The results demonstrate differences in the adhesion performance of the investigated bonding systems for joining the EPDM-based thermal insulation to the metal substrate.

Table 6. Rubber-metal peel strength of the EPDM-based thermal insulation system

sample code	L (mm)	F_{max} (N)	F_{avg} (N)	$F_{avg/L}$ (N/cm)
1-1 T-C6	32.55	18.40	16.37	5.03
1-2 T-C6	32.29	5.40	4.10	1.27
2-1 T-C3	32.68	12.90	12.43	3.80
2-2 T-C3	32.38	11.10	9.07	2.80
T-MC	31.84	49.90	9.64	15.99

The results of the rubber-rubber shear test (Table 7) showed that specimens T-C3-T (2-1) and T-C3-T (2-2) exhibited the highest shear stresses, reaching 0.232 N/mm² and 0.207 N/mm², respectively. During testing, elongation and tearing of the rubber were observed, indicating good adhesion and high mechanical resistance of the bonded joint. In contrast, specimens T-MC-T (3-1) and T-MC-T (3-2) exhibited lower shear stresses, accompanied by interfacial debonding, which indicates weaker adhesion compared with the systems based on C3 and C6 cements.

Table 7. Rubber-rubber shear strength of the EPDM-based thermal insulation system

sample code	L (mm)	b (mm)	A (mm^2)	F_{max} (N)	F_{avg} (N)	F_{max}/A (N/mm^2)
1-1 T-C6-T	21.35	21	448.35	53.25	53.1	0.119
1-2 T-C6-T	21.71	21	455.91	84.9	80.94	0.185
2-1 T-C3-T	22.30	21	468.3	108.8	108.01	0.232
2-2 T-C3-T	22.35	21	469.35	97.10	95.70	0.207
3-1 T-MC-T	21.41	21	449.61	76.8	72.61	0.171
3-2 T-MC-T	21.43	21	450.03	68.89	68.41	0.153

The rubber-metal shear test results (Table 8) showed similar shear stress values for specimens T-C6 and T-C3, amounting to 0.287 N/mm² and 0.240 N/mm², respectively. These results indicate good adhesion between the EPDM-based thermal insulation system and the steel substrate. For specimen T-C6, failure occurred through rubber tearing rather than debonding at the interface, suggesting that the adhesive bond strength exceeded the cohesive strength of the rubber.

Table 8. Rubber-metal shear strength of the EPDM-based thermal insulation system

sample code	L (mm)	b (mm)	A (mm^2)	F_{max} (N)	F_{avg} (N)	$F_{max/A}$ (N/mm^2)
T-C6	21.83	21	458.43	131.5	103.47	0.287
T-C3	21.31	21	447.51	107.3	105.18	0.240

Both specimens, T-MC-T (1-1) and T-MC-T (1-2), exhibited high shear stress values (Table 9). The slightly higher value obtained for specimen T-MC-T (1-1) indicates good homogeneity of the bonded region and efficient crosslinking between the two rubber layers. The obtained results confirm the ability of the MC adhesive to produce mechanically stable joints between the rubber layers.

Table 9. Shear strength of bonded rubber layers in the EPDM-based thermal insulation system

sample code	L (mm)	b (mm)	A (mm^2)	F_{max} (N)	F_{sr} (N)	F_{max}/A (N/mm^2)	
1-1 T-MC-T	27.12	3.35	90.85	75.30	75.30	0.829	0.793
1-2 T-MC-T	25.83	3.35	86.53	65.60	65.50	0.758	

3.3. Application and Dimensional Verification of the Thermal Insulation Lining

The developed EPDM-based thermal insulation system was successfully applied to the inner surface of the steel motor case using the selected adhesive layer TG-HD-727/C3, TG-D-727/C6 cement and thermal insulation compound TG-H-727/3. The application process included surface preparation by sandblasting, adhesive treatment of the chamber wall, positioning of the insulation layer (Figure 3a), air-assisted blowing (Figure 3b), and subsequent vulcanization in an autoclave at 150 °C and 0.5 MPa for 40 min [8].

Particular attention was paid to achieving a uniform insulation thickness and reliable bonding between the insulation layer and the metallic substrate. The use of a duplicated insulation structure consisting of a 0.6 mm adhesive layer and a 1.4 mm to 1.6 mm thermal insulation layer enabled stable positioning of the material and prevented displacement during processing. The selected joining procedure, including a 45° scarf joint and pressure-assisted consolidation, ensured continuous contact between adjacent insulation segments and minimized the possibility of local defects or air entrapment.



Figure 3a.



Figure 3b.

Figures 3 (a and b) Application process of the EPDM-based thermal insulation lining to the rocket motor case

Dimensional verification performed after application of the adhesive layer and thermal insulation confirmed that the final coating thickness was within the target range of 1.8–2.2 mm throughout the chamber length. The measured inner diameter values demonstrated good process repeatability and indicated that the insulation layer was distributed uniformly over the entire internal surface, as shown in Figure 4. Maintaining dimensional tolerances is particularly important in solid rocket motors because local thickness variations may affect thermal protection efficiency, combustion chamber geometry, and subsequent bonding of the liner and propellant.

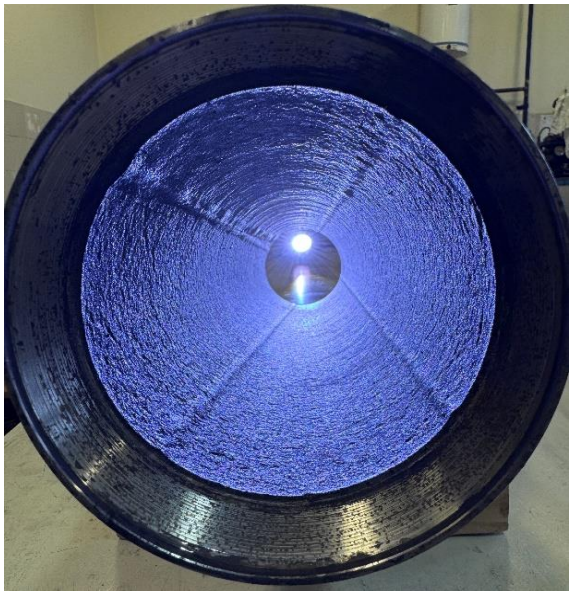


Figure 4. Interior view of the rocket motor case after application of the EPDM-based thermal insulation lining

4. CONCLUSION

An EPDM-based thermal insulation system for application to the inner wall of a steel rocket motor case was successfully developed and verified. The selected thermal insulation compound TG-H-727/3 exhibited satisfactory thermal stability, as confirmed by STA, and was therefore considered suitable for further application studies.

Adhesion testing demonstrated the effectiveness of the developed bonding system. The incorporation of the TG-HD-727/C3 adhesive layer and TG-D-727/C6 cement improved the adhesion between rubber layers and metal case. Satisfactory adhesion between the insulation system and the metal substrate was confirmed through peel and shear testing.

A technological procedure for surface preparation, application of the adhesive system, installation of the thermal insulation lining and autoclave vulcanization was successfully established. The developed procedure enabled the production of a continuous insulation lining with a thickness of 1.8–2.2 mm on the inner wall of the rocket motor case.

The successful lining and vulcanization of steel rocket motor case confirmed the applicability of the developed manufacturing technology under industrial processing conditions. The obtained results demonstrate that the proposed EPDM-based insulation system and the associated application technology are suitable for implementation in rocket motor thermal protection systems.

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