



24th COBEM - 2017



24th ABCM International Congress of Mechanical Engineering
December 3-8, 2017, Curitiba, PR, Brazil

COBEM-2017-1706

EXPERIMENTAL STUDIES OF HEAT INSULATION MATERIALS FOR HYBRID PROPELLANT ROCKET MOTORS

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Abstract. *The ablation rates of non-reinforced epoxies, polyesters, polyurethanes and silicones were estimated experimentally for application as an internal heat insulation of low-thrust hybrid propellant motor. It was established that silicon elastomer has minimal ablation rate in the range 0.33 – 0.52 mm/s depending on conditions of testing.*

Keywords: *hybrid propulsion, heat insulation, ablatives, ablation rate*

1. INTRODUCTION

The low-thrust hybrid propellant test-motor have been developed by the University of Brasilia (Andrianov, et al., 2015) to study applicability of hybrid rocket propulsion as a propulsive decelerator (retro-rocket) in re-entry capsule SARA (in process of development by Aeronautics and Space Institute, Brazil). Operation time of the motor with paraffin grain is near 12-15 seconds. However, the test-motor was designed to operate with other fuels, such like polyethylene, with burning time up to 40-50 seconds. A necessity has appeared to improve heat insulation responsible for thermal protection of structural casing of the hybrid propellant test-motor.

The recent review (Natali, et al., 2016) summarizes fifty years of research efforts on materials for various thermal protective systems. For the mentioned task, materials used for internal thermal protection of propulsion devices, such like liquid and solid rocket motors, are of the most interest. Among them are fiber reinforced polymeric ablatives and heat shielding materials classified as rigid and elastomeric polymeric ablatives. These materials are based on various charring and non-charring polymer matrices, such like thermosetting resins (epoxy, phenolic) or thermoplastic polymers (polytetrafluoroethylene), elastomers (silicones and various rubbers: nitrile butadiene, ethylene propylene diene monomer). In addition to mentioned materials, Tazua (1993) provides insulation characteristics of polyester and polyurethane based cast inhibitors for solid propellant grains, Ahmed (2009) refers to application of carboxy-terminated polybutadiene, copolymer of styrene and butadiene, butadiene-acrylic acid polymer as insulator in solid rocket motors. Some ablative properties of materials whose thermoset or thermoplastic nature depends on cure (or postcure) temperatures (polyamide-imide, polybenzimidazole and polyimide) are given in Marks and Rubin (1969).

There is insufficient information on behavior of ablative polymeric composites of thermal protection in hybrid-propellant motors (Schmidt, 1969). In spite that the latter reference is fifty years old, there are practically no studies on ablative materials for hybrid propulsion. In the work of Story and Arves (2007), it is mentioned that there were no burn throughs in hybrid propellant motor with silica phenolic insulation tested by Scaled Composite. Application of Kevlar-filled ethylene propylene diene monomer in 250,000 pound vacuum thrust hybrid booster was reported in Story, et al. (2003), but no information on behavior of the insulator was provided.

According to Mazetti, et al. (2016), low cost is among advantages of hybrid propulsion technology due to simplified design compared to liquid rocket engines and safety issues compared to solid rocket motors. The tendency to keep the low cost of development should be followed to reinforce competitiveness of the hybrid technology. Thus, so-called COTS (commercial off-the-shelf) solutions could be implemented in process of development of heat insulation. As a result, insulation characteristics of low-cost materials available on Brazilian market were experimentally studied and discussed in the work.

Among the insulation materials subjected to experimental tests were low-cost, non-toxic and room temperature cured thermosetting resins and rubbers available on Brazilian commercial market, such like epoxies, polyesters, polyurethanes and silicones.

Silicones were widely used in various thermal protection systems of missile engines and they showed significant advantages over materials based on butyl rubber, polyurethanes, epoxy resins and others (Donskoy, 1996). It was concluded in the study (Koo, et al., 2011) that silicone resin is more thermally stable than phenolic resin, and tests have shown that composite materials on the base of silicon matrix always performed substantially better than composites based on phenolic resin. In the study of thermal shielding efficiency under exposure to electric-arc-heated gas stream, silicone based materials produced the highest performance in comparison with phenolic and epoxy base materials, but only in a moderate range of heating rate and dynamic pressure (Chapman, 1966).

The choice of other materials was mainly guided by low price, accessibility on market, simplified fabrication technology, and finally, by consideration of moderate performance conditions of low-thrust hybrid propellant motor in comparison with large-scale hybrid or solid propellant rockets. For instance, in solid rocket motors (Fakhrutdinov and Kotelnikov, 1987), rubbers without any mineral filler could be used for internal thermal insulation of front closure, where temperature is high, but velocity of combustion gases is moderate (up to 50 m/s).

The paper presents the results of the first phase of work, where the pure polymers (without particulate of fiber reinforcement) were experimentally tested to reduce the nomenclature of matrix-materials for further study of composite insulators. The primary objective of the work is estimation of ablative characteristics of the selected materials.

Ablative characteristics of materials for internal heat insulation can be estimated and subsequently compared between each other by technical approaches classified in three groups: 1) laboratory characterization by various methods, for example, by thermogravimetric analysis, differential scanning calorimetry etc. (Ahmed, 2009); 2) imitation of environment of a steady flow of hot gas provided by an oxyacetylene torch (ASTM, 2008) or electric arc (Chapman, 1966); 3) firing tests at a reduced scale of propulsion system (Truchot, 1988). In the presented work, the latter technical approach was implemented to estimate ablative characteristics of materials in conditions of internal combustion of a liquid oxidizer and solid fuel.

2. MATERIALS

Description of the materials' composition subjected to study is given in Table 1 together with approximate cost. The cost was recalculated in relation to cost of silicone rubber SI-P on the base of retail price for 1 or 2 kg of the component of the same distributor in June 2016.

Table 1 – Materials selected for experimental study

Group	Specimen	Composition	Density, g/cm ³	Relative cost
epoxy	EP-R	bisphenol A diglycidyl ether (DGEBA) with polyamine based hardener	1.04	1.40
	EP-F	bisphenol A diglycidyl ether and Allyl 2,3-epoxypropyl ether with polyamine based hardener	1.12	1.70
polyester	PS-I	isophthalic resin with catalyst on the base of Methyl ethyl ketone peroxide (MEKP) dissolved in dimethyl phthalate	1.14	0.56
	PS-O	orthophthalic resin with catalyst on the base of Methyl ethyl ketone peroxide dissolved in dimethyl phthalate	1.20	0.40
	PS-C	orthophthalic resin and monomer of styrene with catalyst on the base of Methyl ethyl ketone peroxide dissolved in dimethyl phthalate	1.25	0.48
polyurethane	PUR	*polyurethane prepolymer, diethylhexyl phthalate with mixed isomers of toluene diisocyanate and polyol with diethyltoluenediamine	0.98	2.23
silicone	SI-P	combination of polydimethylsiloxanes with inorganic fillers and copper phthalocyanine (hardness Shore A55)	1.17	1
	SI-V	combination of polydimethylsiloxanes with inorganic fillers and copper phthalocyanine (hardness Shore A55)	1.28	1.10
	SI-B	combination of polydimethylsiloxanes with inorganic fillers and copper phthalocyanine (hardness Shore A10)	1.32	0.66

*some of the components are not mentioned since they are trade secrets of the manufacturer

3. METHODOLOGY

The modular hybrid propellant test-motor described in reference (Andrianov, et al. 2015) was adapted to test materials of heat insulation in conditions of operation close to real ones. Specimens of insulation materials were fabricated by casting in steel molds and then were cut to annular sectors 120° as it shown in Fig. 1.

Specimens in form of annular sectors were installed directly in specially designed additional module (Fig. 2) in the aft part of test-motor between post-chamber and nozzle insert, i.e. close to the region with maximum heat flux in liquid propellant thrust chambers and solid propellant rocket motors according to Sutton and Biblarz (2010). By numerical simulations, it was estimated that average velocity of combustion gases in the module are 100–110 m/s and static temperature is 3300 K.

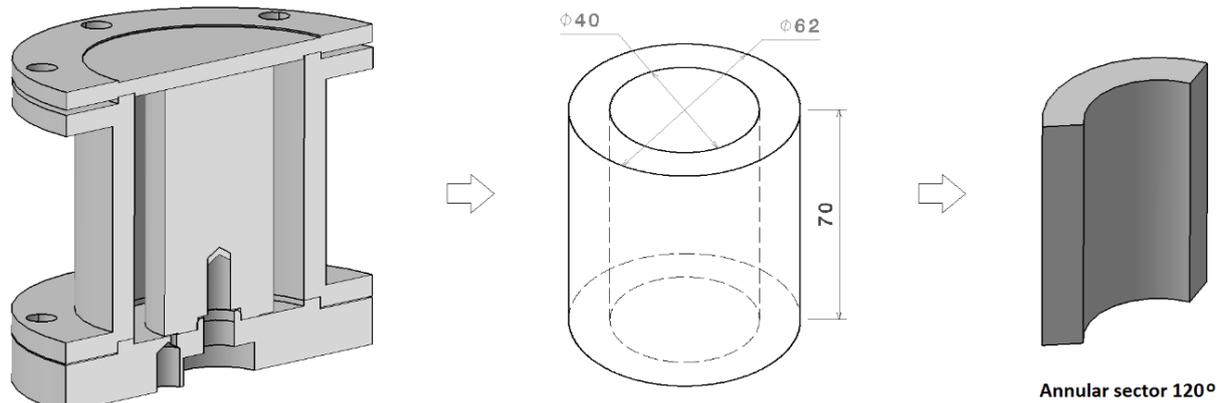


Figure 1. Geometry of heat insulator specimen and casting mold for its fabrication

This module, called specimen compartment, is able to hold three sectoral specimens tightly adjoined along the edges. The specimen compartment is also equipped with nine ports for K- or N-types thermocouples to measure the distribution of temperature on the external surface or inside the specimens during and after the burnout of fuel grain. In the presented report, temperatures were measured principally for rough comparison of thermal diffusivity of tested materials, since for determination of other important parameters, such like velocity of decomposition, the burning time was insufficient.

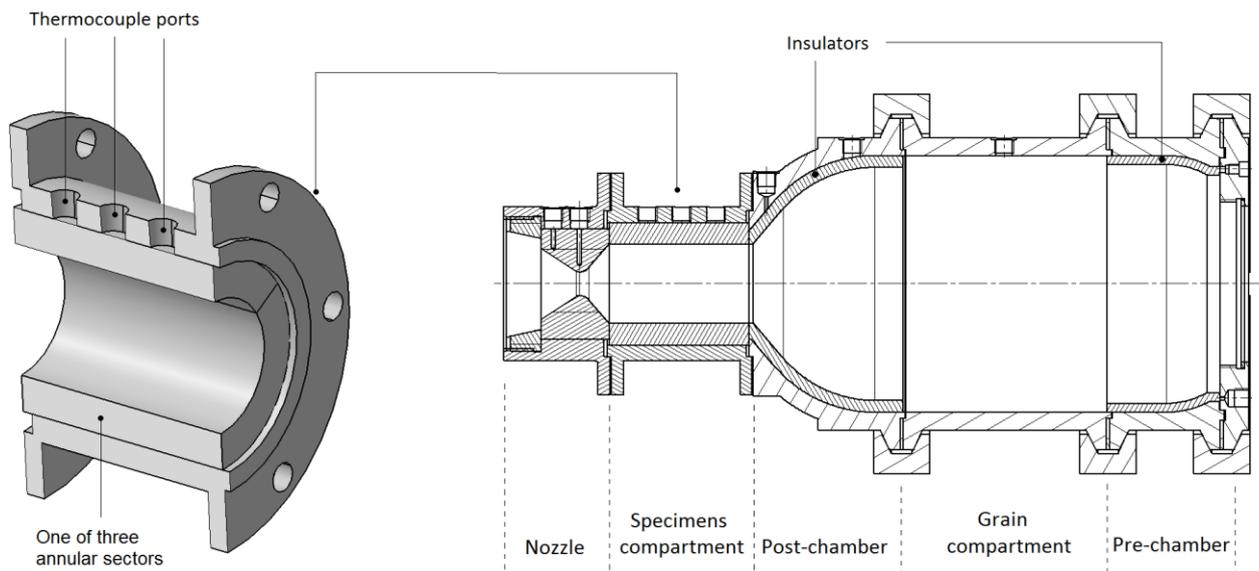


Figure 2. Combustion chamber of test-motor with compartment for specimens of heat insulation materials

There were more than three specimens in compartment for the repetitive tests of ablation study only; in this case, the temperature was not measured and specimens were joined by RTV (room temperature vulcanization) acetic cure silicone sealant that operates at temperatures up to 315°C. Finally, since hybrid motor was installed horizontally, a single test was fulfilled with cylindrical specimen made from one material to study the effect of gravity on gas-dynamic flow. It was assumed in this case that the lower portion of material would burn more than in upper or lateral parts of the cylindrical specimen.

The burning time of motor was limited to the value that would be enough to detect erosion of materials avoiding their complete burning. Thus, the duration of the tests was set for 6 seconds. Liquid nitrous oxide was used as an oxidizer and solid paraffin in form of one-port cylindrical grain as a fuel in all tests. The oxidizer-to-fuel ratio (O/F ratio) was evaluated after every test and its variation was not high from test to test and was in the range 2.2-3.5.

Some of the materials are sensitive to oxidizing environment that is produced by propellant components; for instance, carbon fibres are sensitive to liquid oxygen (Peters, et al., 2000). Thus, two tests with duration of 6 and 10 seconds were fulfilled without injector plate to increase oxidizer flow rate into combustion chamber and to estimate the influence of oxidizer environment on ablation rate of materials. The O/F ratio for these tests were not estimated, because of specifics of straight-flow of oxidizer in combustion chamber. There were no traces of soot on surfaces of paraffin grain after tests that can be regarded as an excess volume of oxidizer in combustion process.

The signals from pressure transducers (installed in oxidizer feeding line and combustion chamber of motor) and thermocouples were recorded and processed by 16-channel data acquisition system.

The thickness and mass of specimens were measured before and after firing of test-motor with the main objective to calculate ablation rates of materials. After testing thin stripes were cut from specimens that had been cleaned from charring and soot. The thickness of specimens (stripes) were measured along their length with step 1 cm, as it shown in Fig. 3.

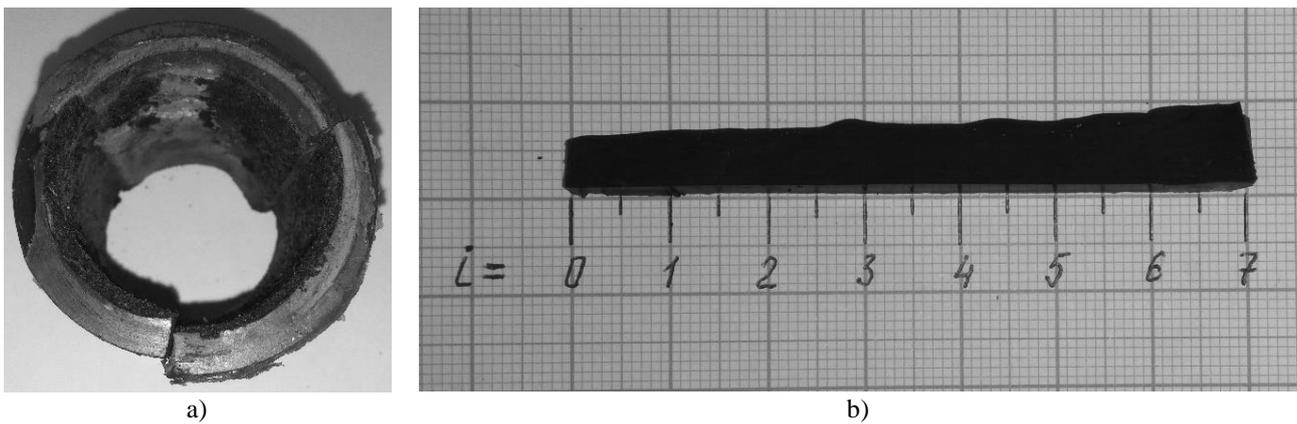


Figure 3. Examination of specimens after burning: a) three annular sectors of specimens extracted from compartment after test, b) i-coordinate grid in centimetres for determination of location of thickness measurement (flow direction is from left to right)

The mean value of remaining thickness t_r for the given specimen was calculated by formula

$$t_r = \sum_i^n \frac{t_i}{n} \quad (1)$$

where i – index that corresponds to coordinate measured from front edge of specimen along its length in direction of gas-dynamic flow (Fig. 3, b), cm;

t_i – the value of remaining thickness at coordinate i , mm;

n – the number of last i -coordinate (equals to 7 for all specimens).

The deviation of specimen's relief from the mean value of remaining thickness was calculated by formula:

$$d_r = \sum_i^n \frac{t_r - t_i}{n} \quad (2)$$

The parameter d_r characterizes the non-uniformity of ablation of specimen along its length.

Since couple of specimens of every material were subjected to testing the mean values of t_r and d_r for every material were calculated by corresponding formulas

$$\bar{t}_r = \sum_j^N \frac{t_{rj}}{N} \quad (3)$$

$$\bar{d}_r = \sum_j^N \frac{d_{rj}}{N} \quad (4)$$

where j – the specimen's index;
 N – the number of tests.

The parameter \bar{t}_r was used for evaluation of insulator material efficiency. The average percentage of ablated layer (by thickness) in relation to its initial thickness was calculated by formula

$$v_t = \frac{t_0 - \bar{t}_r}{t_0} 100\% \quad (5)$$

where t_0 – the initial thickness of specimen (11 mm for all specimens).

The average percentage of ablated layer by mass for the j -th specimen in relation to its initial mass was calculated by formula

$$v_j = \frac{m_{0j} - m_{rj}}{m_{0j}} 100\% \quad (6)$$

where m_0 – the initial mass of j -th specimen, g;
 m_r – the mass of j -th specimen after burning, g.

The average percentage of ablated layer by mass for material was calculated by formula

$$v_m = \sum_j^N \frac{v_j}{N} \quad (7)$$

Finally, the ablation rate by thickness \dot{r} in units [mm/s] and ablation rate by mass \dot{m} in units [%/s] for every material were calculated by the following formulas

$$\dot{r} = \frac{t_0 - \bar{t}_r}{\tau} \quad (8)$$

$$\dot{m} = \frac{v_m}{\tau} \quad (9)$$

where τ – the burning time, s.

For application of obtained results in design process of heat insulation the parameter \dot{r} (calculated from \bar{t}_r) is of great interests. However, the calculation of v_m is more accurate, since the mass of entire specimen was measured before and after test, and measuring of t_r was not done continuously along all length of specimens, but discretely at some selected points. Thus the values of v_t and v_m were compared for the tested materials to understand the expediency and correctness of the adopted methodology for evaluation of important parameter \dot{r} . The discrepancy between average percentage of ablated layer by thickness and mass was calculated by formula

$$d_v = \frac{|v_m - v_t|}{v_m} 100\% \tag{10}$$

4. RESULTS AND DISCUSSION

The effect of gravity on gas-dynamic flow was studied with PUR cylindrical specimen (as it will be shown later PUR is one of the most sensitive materials to ablation). After testing cylindrical specimen was arbitrarily cut into longitudinal specimens-sectors according to the scheme shown in Fig. 4, a. The results of measurement of remaining thickness of every specimen-sector along the cylinder are given in Fig. 4, b and across the specimen – in Fig. 4, c. Calculated characteristics are given in Table 2.

As Fig. 4, b shows, ablation of PUR cylindrical specimen along its length is not uniform. Ablation at *i*-coordinate 1 and 2 cm, i.e. at the entrance of gas-dynamic flow into specimen compartment, is more intensive than at aft part of specimen. It can be explained by generation of turbulent vortexes of combustion gases at the entrance of specimen compartment that enhance the ablation rate. The remaining thickness starts to increase at coordinate *i* = 4 cm or by other words the ablation rate of material decreases slightly probably after stabilization of flow in specimen compartment.

Ablation of cylindrical specimen is not uniform around circumference only at the entrance to compartment (Fig. 4, c), at *i* = 3 cm it stabilizes and contributes to uniform circular cross-section of cylindrical specimen. Thus, the effect of gravity on gas flow has influence on ablation of specimens only at their front portion within bottom sector (150° - 190°). The mean values of remaining thickness are minimal and deviations of specimen’s relief from the mean value are maximal for this sector (Table 2). The sampling from this sector have been avoided in further tests, whose results are given in Table 3.

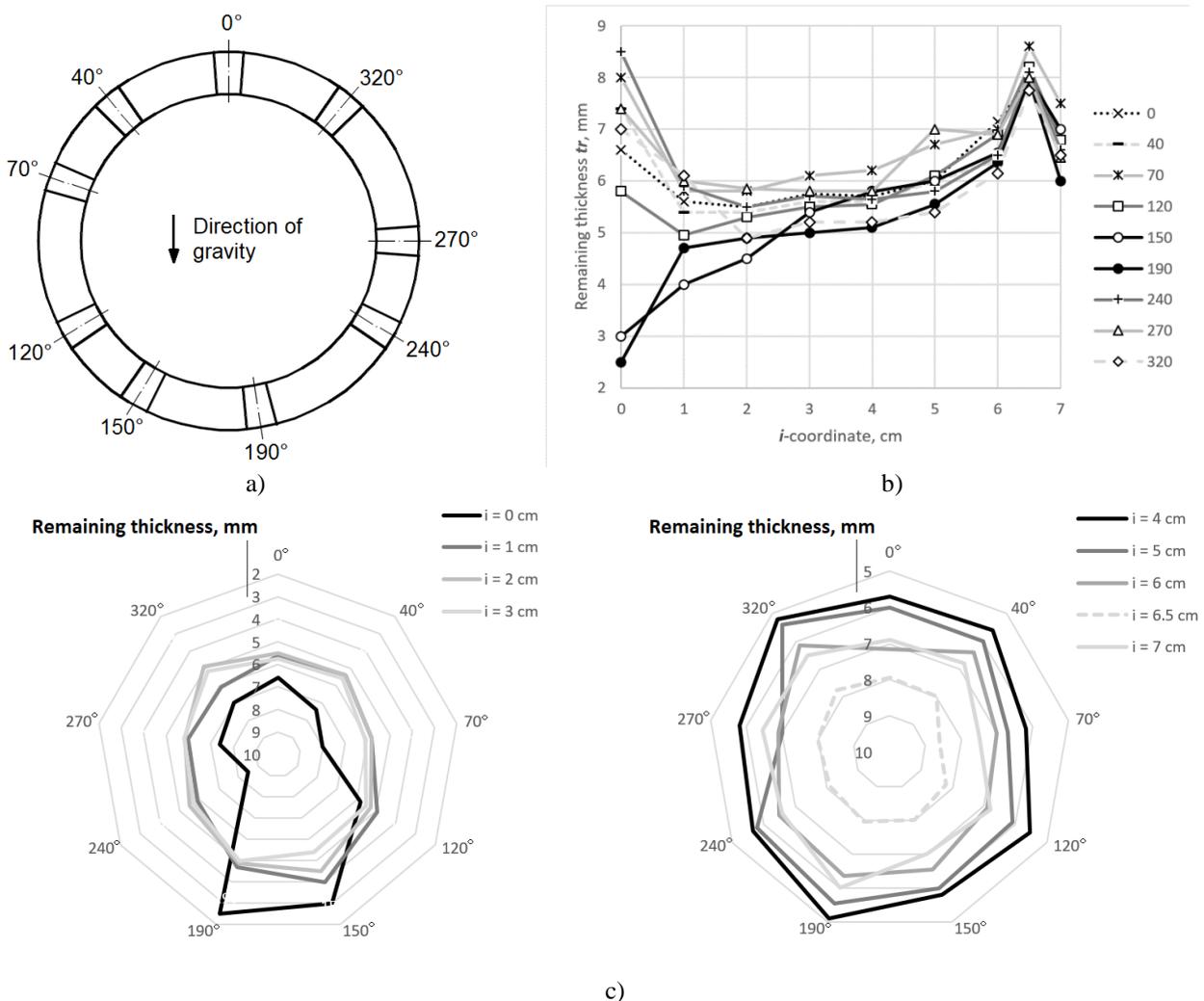


Figure 4. Remaining thickness of cylindrical PUR specimen

Table 2 – Some characteristics for the PUR cylindrical specimen after burning time 6 s

Position of sectors (fig. M3,a)	t_r , mm	d_r , mm	v_t , %	* v_j , %
0°	6.4	0.7	42.3	39
40°	6.3	0.8	42.8	
70°	6.9	0.8	37.7	
120°	6.1	0.8	44.3	
150°	5.6	1.2	49.2	
190°	5.3	1.0	51.5	
240°	6.5	0.8	41.2	
270°	6.6	0.7	40.2	
320°	6.0	0.8	45.3	
Mean values	$\bar{t}_r = 6.2$	$\bar{d}_r = 0.8$	43.8	

*measured for cylindrical specimen

According to these results silicone rubber SI-P has maximal value of remaining thickness, and correspondingly the minimal percentage of ablated layer, that makes this material the most efficient in erosion resistance. In addition, this material is characterized by moderate non-uniformity of ablation. As it was stated above, polyurethane is the most sensitive to ablation among presented materials in the paper. Ablation resistance of epoxy resins are also high in comparison with other materials, whose remaining thickness is less than 7 mm. Epoxy resin EP-F ablates more uniformly along its length together with orthophthalic resin than other materials, however PS-O has lower resistance to ablation. Another advantage of epoxy resins in comparison with silicon rubber is lower thermal diffusivity. It is shown indirectly in the form of plot of temperature versus time for three specimens EP-F, EP-R and SI-P, which were burned together in the same test (Fig. 5). Decrease of temperature at the beginning of burning is a result of cooling of internal surface of specimen by initial flow of oxidizer. After shutdown of motor, the temperature still increases since heat is transferred in the direction of decreasing temperature.

The comparison of average percentage of ablated layer by mass and by thickness is another criterion to evaluate non-uniformity of ablation over specimens' surface. As more closer the values of v_t and v_m , as more uniform is ablation of specimens and more reliable values of ablation rate by thickness could be determined. The average percentage of ablated layer by mass for SI-P is 10% less than for EP-F. The discrepancy for SI-P has the highest value and the same parameter for EP-F – the lowest one. High values of discrepancy for SI-P could be due to imperfection of the chosen method for thickness evaluation; however, it is necessary to note that v_t is greater than v_m for most specimens. It means that the value of ablation rate \dot{r} is overestimated and it can be considered as part of safety factor in process of evaluation of insulator's thickness.

The data from Table 3 was used to calculate ablation rates that are presented in the form of a histogram in Fig. 6 for the purposes of comparison.

Table 3 – Measured characteristics of the tested materials after burning time 6 s at the range of O/F ratio 2.2-3.5

Material	Mean value of remaining thickness \bar{t}_r , mm	Non-uniformity of ablation \bar{d}_r , mm	The average percentage of ablated layer		Discrepancy between v_t and v_m , %
			by thickness v_t , %	by mass v_m , %	
EP-F	7.6	0.3	31.1	31.3	0.9
EP-R	7.3	0.5	33.6	28.5	17.9
PS-C	6.4	0.7	41.5	32.0	29.7
PS-I	6.9	0.4	37.5	33.6	11.9
PS-O	6.7	0.3	38.8	35.5	9.4
PUR	5.7	0.6	48.3	43.8	10.4
SI-B	5.9	0.9	46.3	42.6	8.6
SI-P	7.9	0.5	28.2	20.5	37.4
SI-V	7.2	0.7	34.3	30.9	10.9

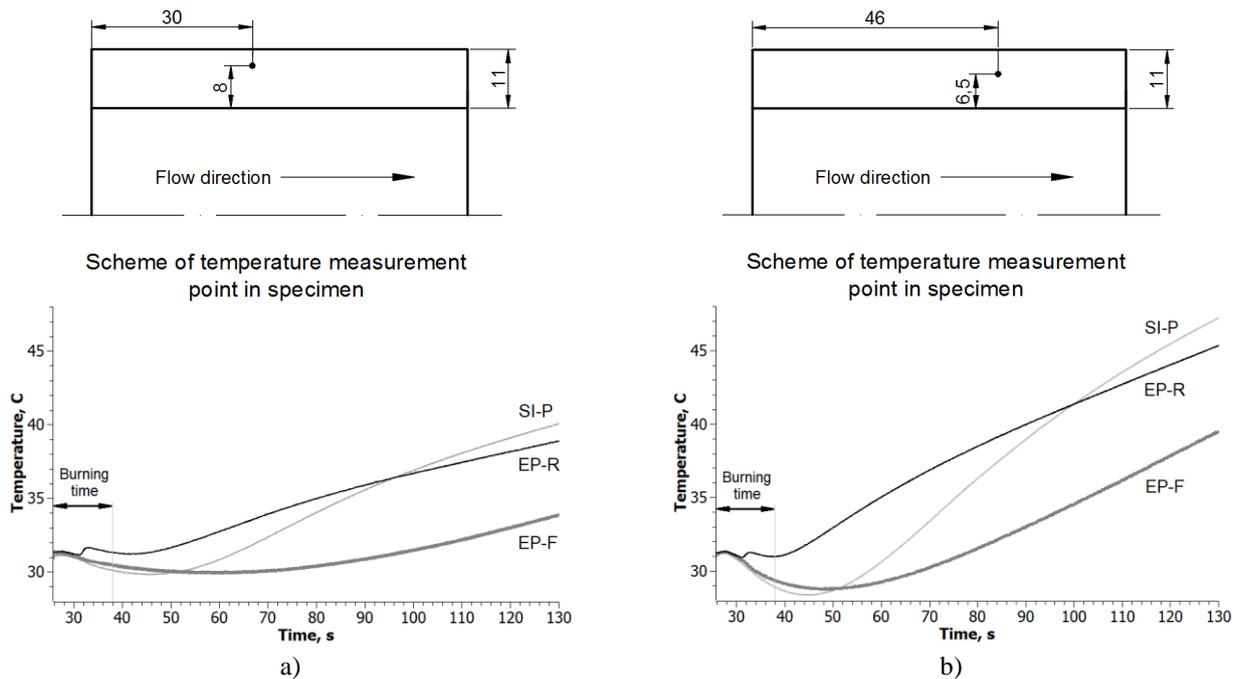


Figure 5. Data on temperature measurements in epoxy resin and silicon rubber for the depth of thermocouple from internal surface of specimen: a) 8 mm; 2) 6.5 mm.

The ablation rate of most resistant to erosion material SI-P is 0.52 mm/s or 3.4 %/s by mass. Thus, it is required from 21 to 29 s to ablate completely 11-mm-thick specimen of SI-P at the same conditions as in presented experiments. It is necessary to note that these values of ablation rate are quite high, since they are very close to values of regression rate of some solid fuels used in hybrid propulsion. For instance, the regression rate is less than 1 mm/s for hydroxyl-terminated polybutadiene with gaseous oxygen at mass flux up to 100 kg/m²·s and less than 0.75 mm/s for low-density polyethylene with hydrazine peroxide at mass flux up to 300 kg/m²·s (Greatrix, 2009).

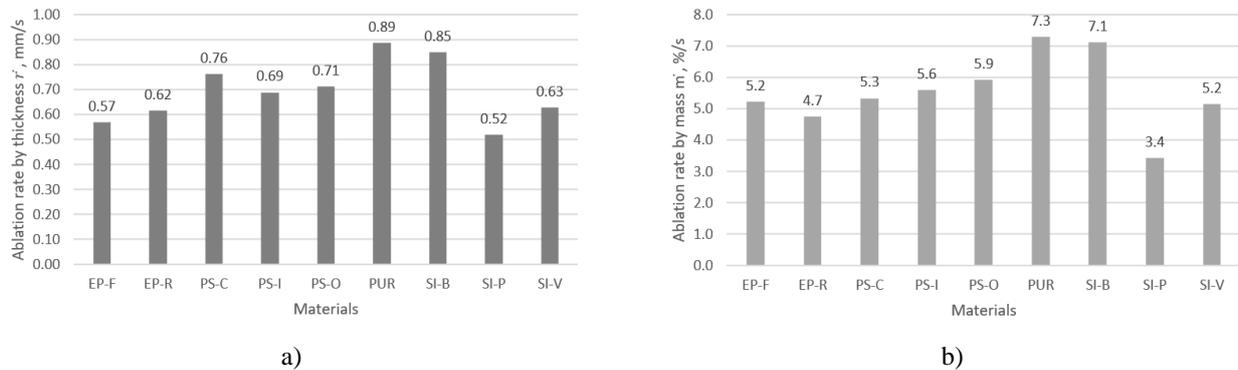


Figure 6. Ablation rates of materials after 6 s tests with O/F ratio 2.2-3.5: a) by thickness, b) by mass

Values of ablation rates determined from the tests with excess of oxidizer are given in Fig. 7. Here, ablation rates by thickness are a bit higher for prolonged 10 s test for most of materials. Thus, ablation rate is not constant during burning. It can be explained by significant influence of transient period at the beginning of burning, which is probably related to heat capacity of materials and their hardness. The unheated layers of material retain their hardness for initial period of combustion and ablation rate is small.

The ablation rates by thickness for most of specimens in tests with excess of oxidizer are lower than in tests with low O/F ratio. The same behavior is for ablation rates by mass, but only for 6 s test. No oxidation effect could be concluded, but for 10 s test, some of materials demonstrate higher values of ablation rates by mass in comparison with results obtained in tests with low O/F ratio. A criterion for evaluation of oxidation effect was not established in the study, since the most resistant specimens, such like silicone rubber SI-P and epoxy resin EP-F demonstrated no significant increase of ablation rate in tests with oxidizer excess. The greater values of ablation rate for most of the

specimens in 6 s tests with low O/F ratio as compared to 6 s test with high O/F ratio can be explained by more efficient burning of propellant in the former case.

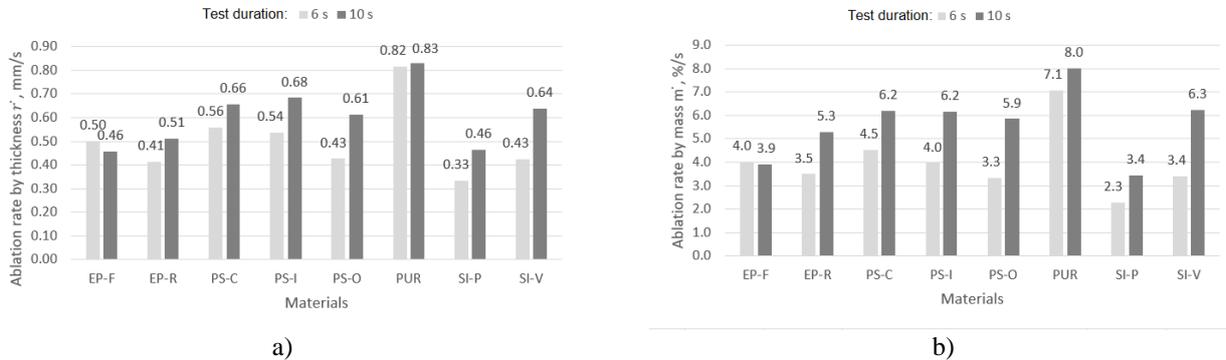


Figure 7. Ablation rates of materials after 6 s and 10 s test with oxidizer in excess: a) by thickness, b) by mass

The results of ablation rate by thickness from Fig. 6 were used to evaluate the thickness of cylindrical heat insulator that ablates completely in 40 s test. The dimensions of the insulator are the same as in Fig. 1, except that the external diameter is variable (depends on evaluated thickness). According to data given in Fig. 8, thicknesses of insulators made from SI-P and EP-F are near 2.1 and 2.3 cm respectively. The mass of the lightest insulator made from SI-P is 0.32 kg.

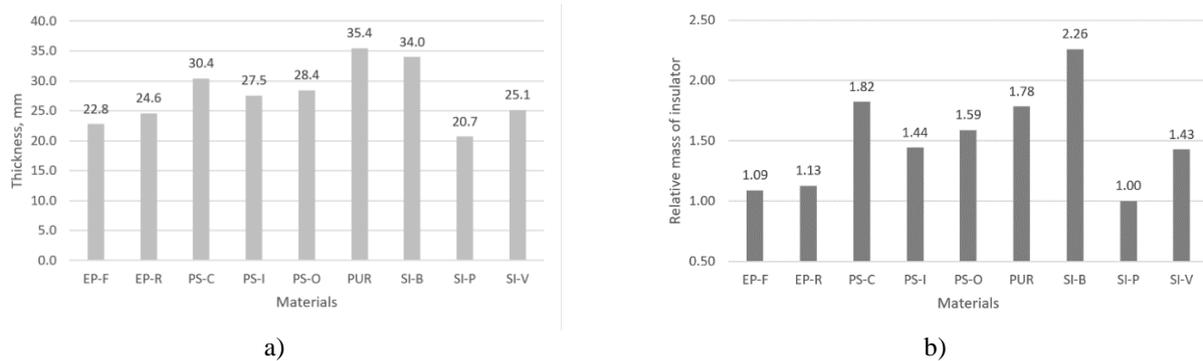


Figure. 8. Evaluation of thickness (a) and relative mass (b) of cylindrical insulator for 40 s test (the mass is related to mass of SI-P insulator)

5. CONCLUSIONS

Ablation rates of various polymeric materials were determined in conditions of firing tests with use of hybrid propellant test-motor. Among tested materials the most resistant to ablation was silicon rubber represented by combination of polydimethylsiloxanes with inorganic fillers and copper phthalocyanine, whose ablation rate was in the range 0.33 – 0.52 mm/s depending on conditions of testing. Polyurethane is the most sensitive to erosion, its ablation rate was in the range 0.82 – 0.89 mm/s.

In summary, averaging results from firings yielded other important conclusions.

1. Specimens ablated non-uniformly along their length, thus the given values on ablation rates were averaged. Nevertheless, the average percentage of ablated layer by mass for some of the materials was less than the average percentage of ablated layer by thickness that slightly overestimates ablation rates given in units of mm/s.
2. Ablation rate for most of the tested materials is not constant during short burning times that probably was caused by influence of transient period of low ablation at the beginning of firing.
3. In horizontally installed test-motor the effect of gravity on gas flow has some influence on ablation of cylindrical specimen within bottom sector where ablation rate is higher than around the specimen. The sampling from this sector has to be avoided.
4. Ablation rates of pure resins and rubbers are very high; their magnitude is of the same order as regression rate of some solid fuels used in hybrid propulsion.

The presented work have shown that the primary obstacle for providing long-time operation of the hybrid propellant motor is not the heat insulating properties of materials but their fast ablation. Thus the matrix materials, which have

shown low ablation rates in the presented tests, have to be reinforced and subjected to further experimental tests with increased burning time.

6. ACKNOWLEDGEMENTS

Authors appreciate the FAP DF (Fundação de Apoio a Pesquisa do Distrito Federal) for financial and administrative support of the work.

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