

## Filled Ethylene-propylene Diene Terpolymer Elastomer as Thermal Insulator for Case-bonded Solid Rocket Motors

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### ABSTRACT

Ethylene-propylene diene terpolymer (EPDM)-based insulation system is being globally used for case-bonded solid rocket motors. A study was undertaken using EPDM as base polymer, blended with hypalon and liquid EPDM and filled with fibrous and non-fibrous fillers. These formulations were evaluated as rocket motor insulation system. The basic objective of the study was to develop an insulation system based on EPDM for case-bonded applications. A series of rocket motor insulator compositions based on EPDM, filled with particulate and fibrous fillers like precipitated silica, fumed silica, aramid, and carbon fibres have been studied for mechanical, rheological, thermal, and interface properties. Compositions based on particulate fillers were optimised for the filler content. Comparatively, fumed silica was found to be superior as filler in terms of mechanical and interface properties. Addition of fibrous filler (5 parts) improved the peel strength, and reduced the thermal conductivity and erosion rate. All the compositions were evaluated for sulphur and peroxide curing. Superior mechanical properties were achieved for sulphur-cured products, whereas peroxide-cured products exhibited an excellent ageing resistance. Rocket motors were insulated with optimised composition and propellant cast, and the motors were evaluated by conducting static test in end-burning mode.

**Keywords:** EPDM elastomer, thermal insulation, solid rocket motor insulators, erosion rate, interface properties, ethylene-propylene diene terpolymer insulation system

### 1. INTRODUCTION

Elastomers are globally used as thermal insulators for case-bonded solid rocket motors<sup>1</sup>. Generally, the solid propulsion system consists of an energetic propellant vital for rocket propulsion and inert subsystems like insulation, liner and inhibition for harnessing the predicted performance characteristics of a solid rocket motor. Each subsystem has a specific role to play and has exact requirements and specifications<sup>2</sup>. The insulator protects the motor case from high-temperature gases and particle streams generated by the

propellant combustion. Insulator should have high-tensile strength and elongation to absorb the mechanical stresses induced to the rocket motor during propellant casting, storage, transportation, and flight. It also helps in anchoring the propellant mass to the inner wall of a rocket motor.

Ethylene-propylene diene terpolymer (EPDM) rubber, which is a terpolymer of ethylene, propylene and a non-conjugated diene, has the highest consumption rate amongst all speciality rubbers in the past three decades. The main attributes of EPDM elastomer are its outstanding resistance to

oxidation, ozonisation, and weathering effects. EPDM rubber has the lowest specific gravity among all rubbers<sup>3</sup>. The most important properties from defence application point of view are its long shelf life and excellent low-temperature properties. Due to these attractive properties of EPDM, it is replacing all other elastomers like nitrile, styrene-butadiene, polydimethyl siloxane, buna-*N*, etc. as insulators in an advanced solid booster such as space shuttle of NASA and inertial upper stage motors<sup>4</sup>. The fillers are incorporated in the elastomeric matrix mainly for reinforcement, lowering of ablation rate as well as to introduce fire retardancy. Silica is mainly used as ablative and reinforcing filler. Aramid fibres have been incorporated in the polymer blends because of their high-thermal capacity and chemical stability, fire resistance, low-thermal conductivity, high-ablation resistance and high-physical adhesion.

Large amount of work has been carried out on rocket motor insulation based on filled and unfilled rubbers and plastics. Nitrile rubber<sup>5</sup> is being used as insulator for the Indian Space and Defence Programmes but suffers from drawbacks like limited-shelf life, relatively higher density, higher-erosion rate and poor low-temperature performance.

Harvey<sup>6</sup>, *et al.* have reported hydrophobic silica, i.e., coated silica-reinforced EPDM insulator. But, it has limitation of higher density (1.10–1.17 g/cc). Liles<sup>7</sup> has patented EPDM-based insulation system reinforced only with polyaramid pulp, which results in insulation having very low-elongation (10–60 %). Further, homogeneous distribution of aramid fibres was difficult to achieve and resulted in nonuniform ablative rates.

Gulliot<sup>8</sup> has studied the reinforcement of carbon fibres in EPDM matrix using solvent processing technique. In solvent processing technique, removal of solvent is difficult which ultimately leads to blister formation in the insulator. Further, dispersion of carbon fibres requires the use of low-viscosity liquid EPDM (80–100 parts) which drastically reduces the mechanical properties of the insulator.

Hence, a systematic research work was undertaken using a medium Mooney viscosity EPDM as primary polymer with ethylene norbornene (ENB) as a third

monomer. Hypalon and low-viscosity EPDM were used as secondary polymers filled with a combination of particulate and fibrous fillers. All the formulations were completely characterised for Mooney viscosity, mechanical properties, interface characteristics and thermal behaviour. Efficacy as rocket propellant insulator was studied by performance evaluation of experimental test motors in end-burning mode.

## 2. EXPERIMENTAL PROCEDURE

### 2.1 Materials

Ethylene-propylene diene terpolymer rubber (H512) with a medium Mooney viscosity (~ 55 MU) having ethylene:propylene (E:P) ratio of 68:32 obtained from the Unimers (I) Ltd, Mumbai, was used as a primary polymer. Hypalon40 (chlorosulphonated polyethylene) from the Dupont Dow Elastomer, USA, of Mooney viscosity (36.7 MU) and liquid EPDM (trilene77) from the Uniroyal, USA, with Mooney viscosity (20 MU) were used as secondary polymers. Precipitated silica (ultrasilVN3) with surface area (175 m<sup>2</sup>/g) and fumed grade silica powder of particle size (12 µm) with surface area of (200 m<sup>2</sup>/g) procured from the Degussa AG, Belgium, were used as reinforcing filler. Twaron fibre from the Teijin, Germany, with fibre length (1–2 mm) was used as polyaramid pulp and PAN-based high-strength carbon fibre from the Nikunj Eximp Pvt Ltd, Mumbai, were used as fibrous filler in the insulator formulations. Sulphur-thiuram accelerator system, from the Bayer (I) Ltd, Mumbai, was used for curing. Di-cumyl peroxide (Di-CUP) from the Perkadox, The Netherlands, as a curing agent and SARET633 from the Sartomer, Pennsylvania, as a coagent were used for peroxide-curing system.

### 2.2 Description of Process

Normally higher-ethylene content and medium viscosity EPDM rubbers are being used for insulator applications<sup>9</sup>. The secondary polymer, hypalon40, was blended with a primary polymer to give polarity to the insulator matrix. Liquid EPDM was used as secondary polymer for dispersion of fibrous fillers like polyaramid pulp and carbon fibres. Batches were compounded in two stages. In stage I, master batch mixing was carried out in Banbury mixer of

capacity (1.5 l) with the rotor RPM77. Master batch mixing consists of mastication of polymers with fillers and additives following predetermined sequence installments. Total mixing time was 10-11 min and in stage II, the master batch was accelerated in a two-roll rubber-mixing mill. Compounded EPDM with sulphur curatives was vulcanised using hand press at 160 °C for 20 min. In case of peroxide curing, vulcanisation was carried out at 170 °C for 10 min.

### 2.3 Characterisation

Cured insulator sheets obtained were characterised for the mechanical properties using Hounsfield, UK Universal tensile testing machine. Process parameters were obtained using a Monsanto Mooney viscometer MV2000 and Monsanto oscillating disk rheometer. An ODR2000 was used to determine the cure characteristics of the EPDM batches. Thermal analysis of pure polymer and insulator samples were carried out in static air at the heating rate of 10 °C/min with alumina as reference standard using Mettler Toledo Star system.

Interface properties such as adhesion between insulator and propellant were measured by 90° peel using wheel peel method in Universal tensile testing machine. Erosion rate measurements were carried out by Plasma Arc Jet Facility and thermal conductivity using a heat flow Mettler thermal conductivity tester model TCHM-DV of R/R Co,

USA, available at the Vikram Sarabhai Space Centre, Thiruvananthapuram. Scanning electron microscope (SEM, Quanta, The Netherlands) was used to study the uniform dispersion of fillers in EPDM.

## 3. RESULTS AND DISCUSSION

### 3.1 Vulcanisation of Compounded EPDM

The basic reaction mechanisms for crosslinking of elastomer by sulphur and peroxide have been discussed quite extensively<sup>10</sup>. In the present study, mercapto benzothiazole (MBT) and tetramethyl thiuram disulphide-ultra-fast accelerator (TMTD) are used with crystex sulphur (polymeric sulphur) and zinc oxide-stearic acid as activators.

The characterisation of vulcanisation process was carried out by ODR2000 and Mooney Scorch in MV2000. The results of rheological properties of sulphur and peroxide-cured products are summarised in Tables 1 and 2, respectively. The Mooney Scorch value ( $MS_{1+4}$  @ 100 °C) for sulphur-curing system of all the formulations ranges from  $t_5$ , m:m = 10 to 14 and  $t_{35}$ , m:m = 16 to 22. The  $t_5$  (Scorch time) value indicates that all the sulphur-cured formulations have sufficient time to permit mixing, shaping, forming, and flowing in the mould before vulcanisation. Further, the data shows that adequate Scorch safety is available for processing temperatures up to 100 °C. Normally, the processing temperatures in the present study are well below 100 °C (around

Table 1. Rheological properties for sulphur curing

Property	EPDM + Silica	EPDM + Aerosil	EPDM + Twaron	EPDM + Carbon
<b>Mooney Scorch (<math>MS_{1+4}</math> @ 100 °C)</b>				
$t_5$ , m:m	14:04	10:47	11:52	10:35
$t_{35}$ , m:m	22:19	16:59	16:07	17:17
<b>Oscillating disk rheometer (ODR @ 160 °C)</b>				
ML [minimum torque (lb)]	8.86	13.09	11.84	10.94
MH [maximum torque (lb)]	34.99	41.39	48.84	44.59
$t_{s1}$ , m:s	2.29	1.97	2.22	1.99
$t_{s2}$ , m:s	2.58	2.22	2.41	2.20
$t_{s10}$ , m:s	2.73	2.39	2.62	2.40
$t_{s50}$ , m:s	5.99	5.53	6.90	6.44
$t_{s90}$ , m:s	18.47	17.29	19.30	18.44

**Table 2. Rheological properties for peroxide curing**

Property	EPDM + Silica	EPDM + Aerosil	EPDM + Twaron	EPDM + Carbon
<b>Mooney Scorch (MS<sub>1+4</sub> @ 100 °C)</b>				
$t_5$ , m:m	9:15	5:42	5:55	6:21
$t_{35}$ , m:m	68:48	68:14	—	—
<b>ODR @ 160 °C</b>				
ML (lb)	8.89	10.72	9.50	7.38
MH (lb)	42.10	44.47	48.97	44.19
$t_{s1}$ , m:s	0.66	0.52	0.71	0.52
$t_{s2}$ , m:s	0.76	0.61	0.81	0.63
$t_{s10}$ , m:s	0.88	0.70	0.97	0.78
$t_{s50}$ , m:s	1.93	1.52	2.20	2.02
$t_{s90}$ , m:s	3.95	2.97	4.55	4.38

80–90 °C). The  $t_{35}$  value shows that there is a faster rise from 5 MU to 30 MU of viscosity indicating faster cure rate for sulphur-curing system at 100 °C.

In the study on peroxide curing, dicumyl peroxide (DCP) is used as curative and zinc dimethacrylate as a co-agent. The MS<sub>1+4</sub> @ 100 °C for peroxide crosslinking ranges from  $t_5$ , m:m = 5.42 to 9.15 and  $t_{35}$ , m:m = 68: 48. The value indicates that initial viscosity rise is fast for peroxide crosslinking but the rise in viscosity from 5 MU to 30 MU is slower as compared to sulphur crosslinking. The dramatic difference in  $t_{35}$  data indicates that the decomposition of dicumyl peroxide takes longer time at 100 °C. This is supported by the findings of Peter<sup>11</sup>, who reported that 50 per cent of DCP decompose in 10 min at 150 °C, whereas it takes 60 h at 100 °C. The rate of crosslink formation/vulcanisation and the extent of crosslinking is monitored by ODR measurements at 160 °C. The time required for 90 per cent cure ( $t_{90}$ ) ranges from 17-20 min for sulphur curative at 160 °C and 3–5 min for peroxide curing conforming that DCP is stable under normal conditions but reacts quickly when exposed to typical vulcanisation temperature.

### 3.2 Particulate Fillers and their Percentages

An important feature of ethylene-propylene rubbers is that these can be highly loaded with fillers and extender oils without adversely affecting

the desirable vulcanisate properties<sup>12</sup>. Silica is amorphous and consists of silicon and oxygen atoms arranged in tetrahedral structure of a three-dimensional lattice with the particle size ranging from 1-30 nm and surface area from 20-300 m<sup>2</sup>/g. Addition of silica to a rubber compound offers a number of advantages such as improvement in tear strength, reduction in heat build-up, and increase in compound adhesion<sup>13</sup>. The tear strength values for the present formulations are 45-55 kg/cm. Further, fumed grade of silica provides thixotropic, anti-settling, anti-blocking and true reinforcement<sup>14</sup>. The results of Mooney viscosity, mechanical properties and interface properties have been reported in Tables 3 and 4.

The compound Mooney viscosity was measured from MV2000 as a characterisation for processability. The viscosity values range from 76, 83, and 97 MU for particulate silica, and 83, 97 and 116 MU for fumed silica formulations corresponding to 30, 35 and 40 parts of particulate filler. These values clearly indicate that with the increase in the percentage of filler, viscosity enhances and hence processing like extrusion or moulding becomes difficult. In view of the above, particulate filler (35 parts) were selected as optimised filler content. As the amount of particulate filler increases, tensile strength increases from 165-186 kg/cm<sup>2</sup> for precipitated silica and 180-234 kg/cm<sup>2</sup> for fumed silica with marginal effect on elongation. The data clearly indicates the increasing trend of reinforcing effect of silica on rubber matrix. Fumed silica shows higher-reinforcing effect as compared to precipitated

**Table 3. Effect of silica on mechanical and interface properties**

Property	EPDM + Hypalon + Silica		
	30 PHR	35 PHR	40 PHR
Mooney viscosity (ML <sub>1+4</sub> @ 100 °C)	76	83	97
Hardness, shore A	67–68	72–73	74–75
Density (g/cc)	1.07	1.08	1.09
Tensile strength (kg/cm <sup>2</sup> )	165.00	177.00	186.00
Elongation (%)	950.00	970.00	989.00
300 % Modulus (kg/cm <sup>2</sup> )	33.00	36.00	34.00
Peel strength (kg/cm)	1.20	1.24	1.28

**Table 4. Effect of aerosil on mechanical and interface properties**

Property	EPDM + Hypalon + Aerosil		
	30 PHR	35 PHR	40 PHR
Mooney viscosity (ML <sub>1+4</sub> @ 100 °C)	83	97	116
Hardness, shore A	74–75	75–76	75–76
Density (g/cc)	1.05	1.06	1.07
Tensile strength (kg/cm <sup>2</sup> )	180.00	185.00	234.00
Elongation (%)	990.00	962.00	1018.00
300 % Modulus (kg/cm <sup>2</sup> )	38.00	45.00	39.00
Peel strength (kg/cm)	1.31	1.35	1.42

silica due to its finer particle size (12  $\mu$ m) and higher-specific surface area (200 m<sup>2</sup>/g). Lower density (1.05-1.07 g/cc) was obtained with fumed silica as against the precipitated silica (1.07-1.09 g/cc). Also, comparatively better peel strength (1.3-1.4 kg/cm<sup>2</sup>) was achieved with fumed silica. Lower- thermal conductivity (aerosil:0.193 W/m K: silica:0.2 W/m K) and erosion rate (aerosil:0.062 mm/s: silica:0.069 mm/s) have been achieved for aerosil-filled formulations. The major advantage of aerosil is that silica particles have silanol groups on their surfaces, while *Si-O-Si* bonds are located inside these particles. This typical structure gives hydrophobic nature to this class of silica and these silanol groups are reactive sites for surface treatment. These free-silanol groups may also help to achieve good interface properties with propellant (1.2-1.4 kg/cm), for all these formulations, though the base polymer EPDM is non-polar in nature. Hence, the fumed grade of silica was selected for further studies on formulations containing fibrous filler.

### 3.3 Incorporation of Fibrous Fillers

The combination of fumed silica with fibrous filler has been incorporated in the insulator recipe because of their very high-thermal capacity, superior-chemical stability, fire-resistance, low-thermal conductance, high-abrasion resistance, and enhanced physical adhesion. The base formulation selected contains fumed silica (35 parts) and optimised quantity of fibrous filler (5 parts). The dispersion of these fibres was very difficult in medium viscosity primary polymer, and secondary polymer like hypalon40. Hence, a low-viscosity liquid EPDM was used as a secondary polymer. The dried fibres were dispersed in liquid EPDM and then blended with primary polymer. Liquid EPDM<sup>5</sup> acts as an adhesion binder to combine solid fillers, polyaramid fibres, and solid EPDM rubber in the insulator recipe. Basically, liquid EPDM has the same chemical structure as the solid EPDM rubber and is completely compatible. The primary and secondary polymers differ in molecular weight

only. These polymers co-cure simultaneously as the diene monomer (ENB) is also the same. The net effect is that the storage security of the rocket during its service is ensured. The results of mechanical and interface properties are summarised in Table 5. The compound Mooney viscosity  $ML_{1+4}$  @ 100 °C for these formulations is also comparatively lower (56-78 MU) because of incorporation of liquid EPDM making it very easy for processing. The tensile properties of Twaron containing insulator range from 150-155 kgf/cm<sup>2</sup> with elongation 625-900 per cent. Similarly, carbon-containing insulator gave tensile strength from 176-180 kgf/cm<sup>2</sup> with very high-elongation from 700-1170 per cent. The data confirms that carbon fibres impart higher mechanical properties due to their reactive functional groups, which help in reinforcement as well as add polarity to the surface<sup>16</sup>. While, Twaron pulp exhibited decreased mechanical properties due to highly aromatic and rigid structure of polyaramid, the higher values of peel strength for Twaron and carbon-filled formulations (1.4-1.57 kg/cm) can be attributed to the polar nature of fibrous fillers. Comparatively lowest density (Twaron-1.04 g/cc: Carbon-1.05 g/cc), lowest thermal conductivity (Twaron-0.173 W/m K: Carbon-0.18 W/m K) and erosion rate (Twaron-0.059 mm/s: Carbon-0.06 mm/s) have been achieved for Twaron and

carbon-filled formulations as compared to silica and aerosil-based formulations.

### 3.4 Studies on Thermal Analysis

The decomposition of pure EPDM, EPDM blends with secondary polymers and fillers shows a single-stage sharp weight loss over a very short-temperature range with a  $T_{max}$  ranging from 479-492 °C (Table 6) with a single decomposition exotherm ranging from 470-485 °C. These observations are in close agreement with those of Kole<sup>17</sup>, *et al.* The high-thermal stability of EPDM and its blends is attributed to its saturated main chain structure<sup>18</sup>.

All the vulcanised insulators showed two-stage weight loss. The decomposition for sulphur-cured samples starts around 220-250 °C and major decomposition occurs between 420-500 °C with  $T_{max}$  ranging from 483-498 °C. The occurrence of two-stage weight loss could be attributed to the presence of volatile matter, eg, stearic acid and by-products of sulphur vulcanisation, which cause initial weight loss around (5-6 %) at 220-250 °C. The second exotherm or  $T_{max}$  shifts to higher temperature for filled vulcanised insulators. The  $T_{max}$  is the highest for peroxide-cured formulations (498.2 °C) indicating better thermal stability of these formulations.

**Table 5. Fibrous filler (mechanical interface properties)**

Property	EPDM + Liquid EPDM			
	Twaron (S)	Carbon (S)	Twaron (P)	Carbon (P)
Mooney viscosity ( $ML_{1+4}$ @ 100 °C)	78	74	60	56
Hardness, shore A	83-84	76-77	79-80	74-75
Density (g/cc)	1.04	1.05	1.05	1.06
Tensile strength (kg/cm <sup>2</sup> )	152.00	176.00	155.00	180.00
Elongation (%)	899.00	1170.00	625.00	700.00
300 % Modulus (kg/cm <sup>2</sup> )	92.00	38.00	55.00	49.00
Peel strength (kg/cm)	1.57	1.40	1.36	1.30
Thermal conductivity (W/m K)	0.17	0.18	0.17	0.18

S is the sulphur and P is the peroxide.

**Table 6. Thermal analysis of pure polymer-vulcanised insulator**

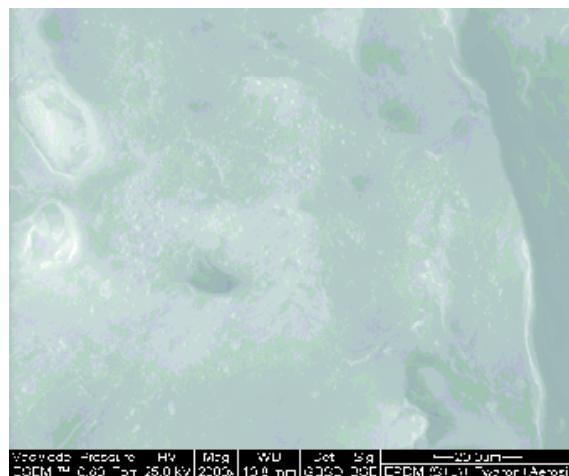
Sample	DTA	TGA	
	Exotherm	Temp range	Wt loss
	(°C)	(°C)	(%)
<b>Pure polymer</b>			
EPDM H512	484	382–492	94.2
<b>Pure polymer + Fillers</b>			
EPDM + Liquid	480	378–487	91.0
EPDM (L.E.) + Twaron			
EPDM+L.E. + Twaron + Aerosil	478	355–488	69.0
EPDM + L.E. + Carbon	470	368–479	92.0
EPDM + L.E. + Carbon + Aerosil	479	375–489	72.0
<b>Vulcanised formulations</b>			
<b>Sulphur cured</b>			
EPDM + Silica	476	198–267	6.6
		380–493	55.0
EPDM + Aerosil	483	181–262	7.0
		378–496	54.0
EPDM + Aerosil + Twaron	486	206–301	5.0
		378–490	60.0
EPDM + Aerosil + Carbon	475	197–252	6.0
		367–483	50.0
<b>Peroxide cured</b>			
EPDM + Aerosil	487	174–278	9.0
		382–498	48.0

### 3.5 SEM Studies

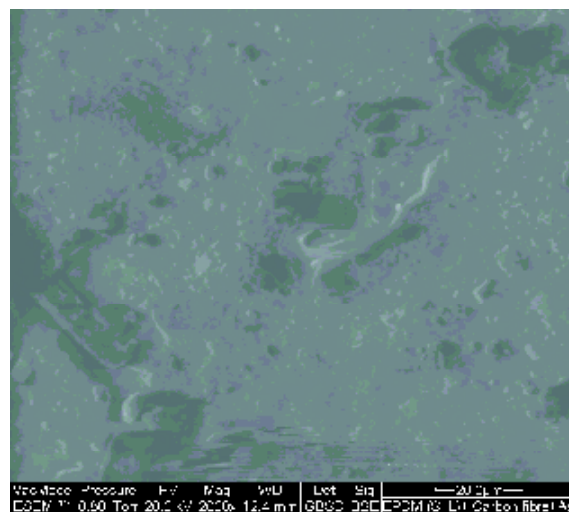
SEM studies were carried out for polymer blend only with fibres and polymer blend with fibre and aerosil. The back-scattered electron (BSE) images are shown in Fig. 1. The back-scattered image clearly shows the uniform dispersion of fibres and particulate filler in the polymer matrix.

### 3.6 Performance Evaluation

Medium size rocket motors (L ~ 200 mm, OD ~ 110 mm) were selected for actual application studies with a typical composite case-bonded propellant formulation. The rocket motors were pre-insulated (~2 mm thick) with EPDM formulation and coated with suitable liner. A typical composite propellant composition based on HTPB-AP-*Al* was cast in these rocket motors. The propellant was cured, x-rayed and subjected to static evaluation in end-burning mode for 20 s.



(a)



(b)

**Figure 1. SEM pictures of filled EPDM: (a) EPDM+L.E. +Twaron+Aerosil and (b) EPDM+L.E.+Carbon +Aerosil.**

A neutral *P-t* profile was obtained. The pressure level was nearly constant (average pressure 47.6 kg/cm<sup>2</sup>) which showed that the bond between propellant-insulator was excellent. The *P-t* profile is shown in Fig. 2. Post-firing observations after extinguishing the motors revealed the presence of semi-charred insulator throughout the motor length. A thin layer (~1 mm thick) of virgin insulator was intact below the char layer. The rise in skin temperature measured at the end of firing was 4–5 °C at the nozzle end (after 20 s of propellant burning), proving the efficacy of the insulator.

MAX	AVRG	AREA	DELAY	TIME	CSTAR/ISP
PRESSURE 58.31 KSC	47.58 KSC	1149.47	30 mS	24.16 s	1650.84
PRESSURE (Tb)	50.33 KSC	1104.64		21.95 s	
TA Dt=8.02 mm	Wt=3.45 kg				

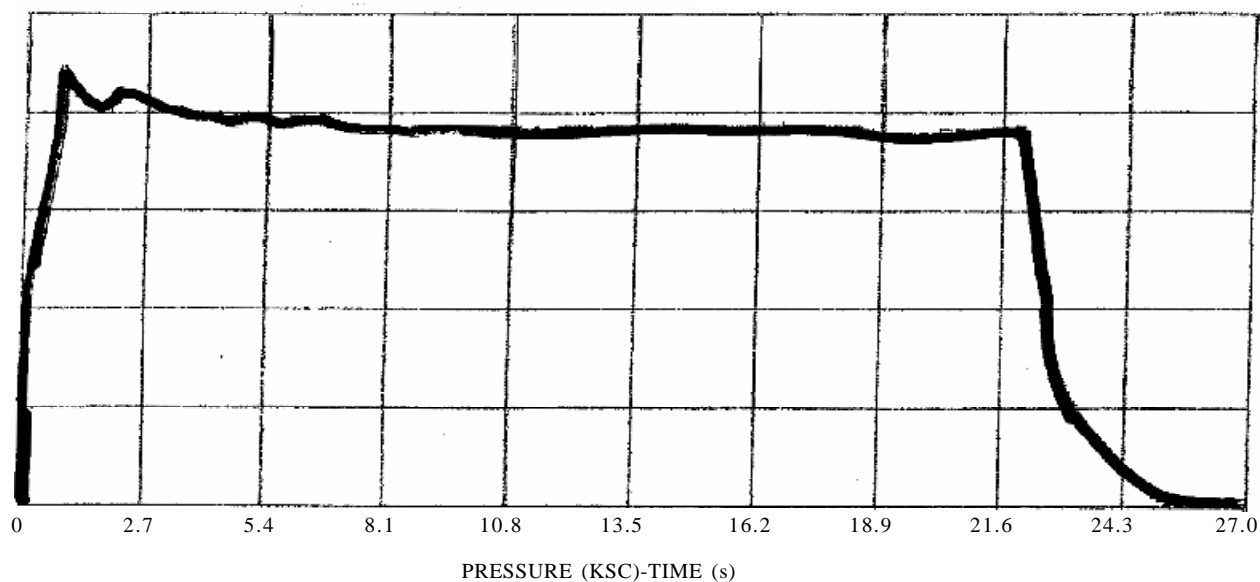


Figure 2. *P-t* profile for insulated solid rocket motor during firing.

### 3.7 Ageing Studies

Ageing studies were carried out at 50 °C for 5 days for two formulations based on Twaron and carbon fibres with sulphur as well as peroxide curative systems. The temperature selected was a curing temperature of a typical case-bonded composite propellant. The tensile strength for sulphur-cured samples increase from 152–160 kg/cm<sup>2</sup> for Twaron and 176–186 kg/cm<sup>2</sup> for carbon with marginal decrease in elongation. In case of peroxide curing, tensile strength increased from 155–157 kg/cm<sup>2</sup> for Twaron and 180–183 kg/cm<sup>2</sup> for carbon with the marginal decrease in elongation. The increase in tensile strength properties could be attributed to slight post-curing at high-temperatures. The negligible variations observed with peroxide-cured samples indicate better stability of vulcanised insulator.

## 4. CONCLUSION

The combination of fibrous and particulate fillers utilises advantage of both the systems and results in increase in the thermal insulation properties leading to a system with low-thermal conductivity, low-erosion rate, reduced inert weight of propulsion

system by reducing the density of insulation without adversely affecting the mechanical properties of the insulation system.

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