

# Erosion of Nozzle Throat Inserts and Silica-Phenolic Ablative Liner Morphology

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A requalification test program for 250-kg thrust nozzle throat inserts (NTI) for the Augmented Satellite Launch Vehicle mission was necessitated due to 6 s of extra burning duration compared to the Satellite Launch Vehicle mission. It has been observed that the erosion of NTI was closely related to chamber pressure, cumulative burning time, and the erosion of silica-phenolic-based ablative liner morphology. This paper presents an analysis of the erosion results of autotranspiration-cooled NTI and silica-phenolic ablative liners used in the combustion chamber.

## Nomenclature

$C_*$	= characteristic exhaust velocity, cm/s
$C_p$	= specific heat of gas (freestream condition)
$D_t$	= throat diameter, mm
$g_0$	= gravitational constant, cm/s <sup>2</sup>
$h_g$	= combustion gas heat-transfer coefficient, cal/cm <sup>2</sup> -s °C
$K$	= constant
$P_c$	= chamber pressure, kg/cm <sup>2</sup>
$P_r$	= Prandtl number (freestream condition)
$r_c$	= throat radius of curvature, mm
$U$	= freestream velocity of combustion, cm/s

## Introduction

THE nozzle throat insert is the most critical component in the attitude control rockets of launch vehicles, since the rate of heat transfer from the products of combustion reaches maximum at the throat, and the constancy of the throat diameter is very critical in maintaining precise thrust and chamber pressures. Hence, the material chosen for the nozzle throat insert must withstand the stringent environmental conditions of high convective heat transfer, high temperature, and possible oxidizing atmosphere of the combustion products. Based on the results of several studies,<sup>1</sup> a molybdenum-based composite was selected as the nozzle throat insert material for the aforementioned application. A schematic diagram of 250-kg thrust bipropellant control rocket thruster assembly is shown in Fig. 1.

Nozzle throat inserts developed for 250-kg thrust bipropellant control rocket thrusters for Indian satellite launch vehicles (SLV) were initially qualified for 23 s (cumulative) and performed quite satisfactorily in all their flights. However, in the case of the augmented satellite launch vehicle (ASLV), an additional requirement of pitch and yaw control of the second stage has resulted in more seconds of cumulative burning, from 18 to 24 s in the SLV mission. Product specifications for the NTI are given in Table 1. Hence, a requalification test

program became essential in order to evaluate the performance of various critical subsystems like the transpiration-cooled nozzle throat insert and the choice of silica-phenolic (Refrasil) ablative laminates as combustion chamber material.

This paper presents the analysis of the indigenously developed molybdenum-based nozzle throat inserts for the ASLV program, and the erosion of the throat diameter of inserts is

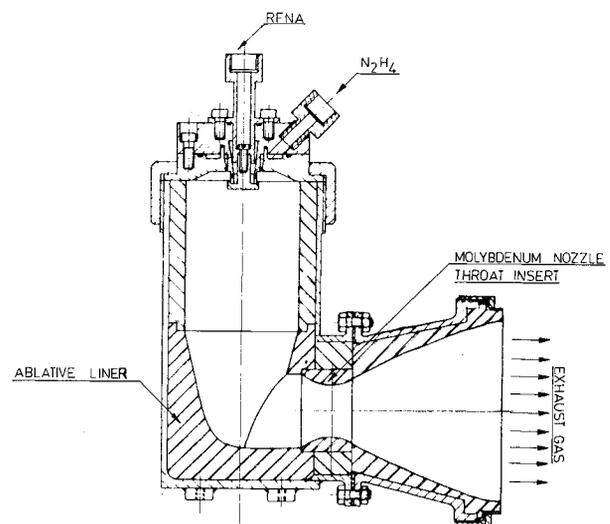


Fig. 1 250-kg bipropellant thruster assembly.

Table 1 Thrust nozzle throat insert product specification, 250-kg

1) Material	: Molybdenum-based composite
2) Hardness (Rockwell-B)	: RB 85
3) Density, g/cc	: 9.8
4) Mass, g	: 325
5) Throat diameter, mm $\phi$	: 30.8 + 0.00 - 0.05
6) Visual inspection	: a) Voids and surface cracks strictly prohibited b) No sharp edge at convergent and throat interface c) Smooth surface finish
7) Radiographic inspection	: a) Voids and cracks prohibited

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correlated with the type of ablative liner, cumulative burning time, and chamber pressure of the motor.

**Theory**

Erosion of the throat inserts depends upon velocity, mixture ratio, chemical reaction between the nozzle throat material and the products of combustion, and impingement on the semisolid viscous composite material.

It is known from the Bartz equation<sup>2</sup> that the heat-transfer coefficient  $h_g$  at the rocket nozzle throat in the absence of coolant injection is

$$h_g = \frac{K}{(D_t)^{0.2}} \left[ \frac{U_\alpha^{0.2} \times C_p}{P_{ra}^{0.6}} \right] \times \left[ \frac{P_c \times g_0}{C_*} \right]^{0.8} \times \left[ \frac{D_t}{r_c} \right]^{0.1} \quad (1)$$

In this particular test program, many parameters, viz.,  $r_c$ ,  $C_*$ ,  $P_r$ ,  $U$ ,  $C_p$ ,  $g_0$ , were held constant. Hence, Eq. (1) can be written as

$$h_g = K_1 \frac{(P_c)^{0.8}}{(D_t)^{0.1}}$$

where

$$K_1 = K \left[ \frac{U_\alpha^{0.2} \times C_p}{P_{ra}^{0.6}} \right] \times \left[ \frac{g_0}{C_*} \right]^{0.8} \times \left[ \frac{1}{r_c} \right]^{0.1} = \text{const} \quad (2)$$

It has also been found that the rate of erosion  $e$  is proportional to  $h_g$ . Equation (2) can, therefore, be rewritten as

$$\begin{aligned} e &= K_2 h_g \\ &= K_3 \times \frac{(P_c)^{0.8}}{(D_t)^{0.1}} = K_3 h_x \end{aligned}$$

where

$$\begin{aligned} K_3 &= K_1 \times K_2 = \text{const} \\ h_x &= \frac{(P_c)^{0.8}}{(D_t)^{0.1}} \end{aligned} \quad (3)$$

if all the other parameters remain unaltered. Similarly, it has been found experimentally that in the case of uncooled ablative liners at lower chamber pressure (0.784 MPa), the formation of charred layer for reinforced plastics is related empirically to the combustion time (firing duration)<sup>3</sup> by

$$\text{Charred depth (in.)} = 0.04 \left( \frac{\text{in.}}{\sqrt{s}} \right) \sqrt{\text{firing duration (s)}}$$

In the case of silica-phenolic composite, it is presumed that ablation of resin continues even at "off" time temperatures, whereas metallic nozzle throat inserts are expected to cease ablating at a much higher temperature.

For the present discussion, the ratio of cumulative burning time to total operating time (i.e., time factor  $t/T$ ) of the motor is taken into consideration for comparing the erosion of the throat inserts.

**Process Details**

**Nozzle Throat Inserts**

High-purity molybdenum metal powder of required particle size was sieved and mixed with paraffin wax, dissolved in carbon tetrachloride, and dried in air. Required amounts of the dried, sieved powder were weighed and compacted at a pressure of 650 MPa to a green density of 60–65% theoretical density. Then the green compacts were dewaxed at 850°C and subsequently sintered in a pit-type hydrogen atmosphere furnace at 1550 ± 10°C for 2 h. Sintered compacts were then impregnated with Cu alloy in a hydrogen atmosphere and machined to the required dimensions. Finally, an oxidation resistance silicon coating was applied on all the finished components by the chemical vapour deposition technique at a temperature of 850°C prior to use.

**Ablative Liners**

Two different types of silica-phenolic composites were used as liner materials during the qualification program.

*1) Random Fiber-Molded Silica-Phenolic Composite (RFM)*

For the processing of random fiber-molded silica-phenolic composite, a desired size (10 × 10 mm) of silica chops were cut from prepreg silica cloth. The chops were then poured into a die, packed under high pressure (6.895 MPa) and heated to resin gel temperature to obtain a very dense composite.

*2) Stacked Silica-Phenolic Composite (SPC)*

In the case of stacked silica-phenolic composite (SPC), silica prepreg cloths were stacked in a mold, pressed at high pressure, and then cured at a resin gel temperature similar to the RFM technique. Composites thus obtained were subsequently machined for fabricating the combustion chamber liner. During molding, care was taken to insure that the silica fibers were oriented perpendicular to the combustion chamber axis to improve the erosion resistance by lowering the ablative regression rate.<sup>4</sup>

**Motor Tests**

Four tests were carried out at sea level for different burning durations and chamber pressures.<sup>5</sup> Thrusters were made with two different types of silica-phenolic composites as ablative liners. Tests 1 and 2 were planned with RFM silica-phenolic composite, and tests 3 and 4 with stacked silica-phenolic composite as the ablative liner.

The propellant injectors (sheet-impinging type) were initially calibrated with respect to flow rate of propellants and generation of required chamber pressure before use in actual material evaluation tests. The propellants, hydrazine (N<sub>2</sub>H<sub>4</sub>)

**Table 2 Ground qualification test results of 250-kg bipropellant thruster with erosion figures**

Test no.	NTI no.	Type of combustion chamber liner	Cumulative burning time, s	Total operation time, s	Average chamber pressure (gauge), MPa	Throat erosion		Remarks
						Profile projection method	Toolmaker's microscope method	
1	03	Randomly molded fiber composite	24.00	77.330	1.534	-1.870	-0.584	Appreciable nonuniform silica deposition at convergent and throat regions
2	02	Randomly molded fiber composite	30.00	95.500	1.642	7.600	8.950	Heavy nonuniform erosion with silica deposit at convergent and throat regions
3	01	Stacked silica-phenolic composite	33.04	80.790	1.364	0.000	0.162	Negligible deposit with very small erosion
4	04	Stacked silica-phenolic composite	32.05	80.300	1.621	1.490	-1.230	Very little silica deposit at throat and convergent regions

Table 3 Mass and diameter variations of nozzle throat insert with different measuring techniques

Test no.	NTI no.	Before test		After test		Difference		% erosion	
		Mass, g	Diam, mm	Mass, g	Diam, mm	Mass, g	Diam, mm	Toolmaker's microscope	Profile projection method
1	03	329.81	30.78	318.80	30.69	11.01	-0.09	-0.584	-1.870
2	02	332.08	30.76	295.50	32.03	36.58	1.27	8.950	7.600
3	01	315.89	30.77	310.50	30.79	5.39	0.02	0.162	0.000
4	04	327.44	30.77	324.40	30.66	3.04	-0.19	-1.230	1.490

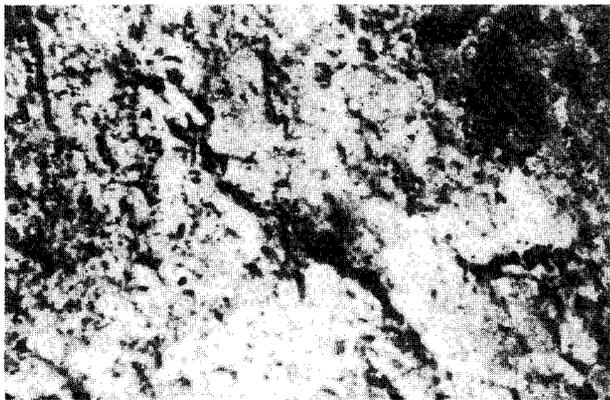


Fig. 2a Stereomicrograph of throat insert surface with heavy silica deposit (mag. X15); liner; RFM.

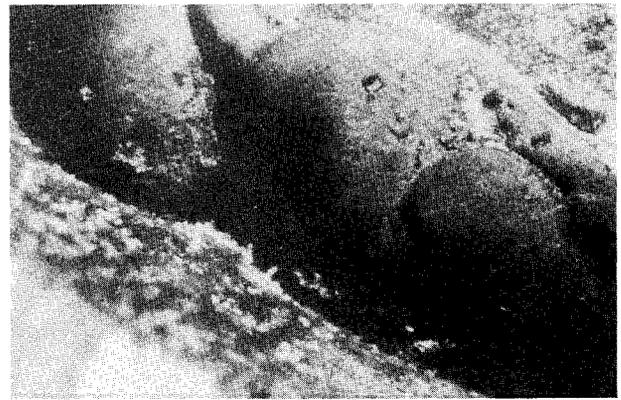


Fig. 2b Stereomicrograph of throat insert surface with heavy local erosion (mag. X15); liner; RFM.

and red fuming nitric acid (RFNA), were stored separately in two stainless steel tanks and pressurized with nitrogen gas (3000 psi).

#### Motor Test Procedure

The injector was fitted with the combustion chamber and connected to the propellant tanks through electrically operated solenoid valves.

During the tests, in which RFNA lead was 3-4 ms, the propellants came in contact with the ablatively lined combustion chamber, and the products of combustion were allowed to pass through the predesigned nozzle to have the desired thrust effect. Test results with measured erosion figures, which will be discussed in the following section, are given in Table 2.

#### Measurement of Throat Erosion

The nozzle throat inserts were removed after firing with utmost care and examined in situ for deposition/erosion. In each test, five readings of the throat diameter were taken and the average was considered for performance evaluation. The readings took into account the deposition/erosion, if any, on the throat. A toolmaker's microscope, with an accuracy of 0.01 mm, was used to measure the throat diameter before and after the firing.

The fired inserts were further examined more precisely by profile projection. Projected throat contours were recorded in each case at a magnification of  $\times 10$  on a tracing paper, and compared before and after firing. The area measurements were carried out by planimeter. The results obtained are given in Table 3.

### Results and Discussions

The aforementioned studies are made with a view to explain the erosion behavior of NTI with respect to 1) heat flux factor ( $hx$ ), 2) time factor ( $tx$ ), and 3) morphology of the ablatively liner system.

In tests 1 and 2, heavy deposition of silica on the convergent and throat regions of throat inserts was observed (Fig. 2a).

The erosion of ablatively liner was greater in test 2 than in test 1. In both cases, the presence of small burning pieces was noticed in the exhaust flame during motor firing.

In these two tests, both time factors and morphology of RFM were similar (Tables 2 and 4), and the only difference was with respect to  $hx$ , which was higher in test 2 than in test 1.

Both experimental results and the theoretical calculations showed that the erosion of the NTI and of the chamber liner was directly proportional to  $hx$ . Though the overall erosion measured by profile projection and the toolmaker's microscope techniques in test 1 was negative, the visual and microstructural examinations revealed heavy positive local erosion of the NTI (see Fig. 2b).

Results of tests 3 and 4, which correspond to stacked silica-phenolic ablatively liner, showed that the major difference was only in regard to  $hx$ , which was higher in test 4. The greater erosion observed in test 4 could again be attributed to the greater  $hx$ .

A comparison of the results obtained with the two types of ablatively liners (tests 2 and 4) clearly showed that erosion was much less with stacked silica-phenolic than with the RFM for similar  $hx$  factors (Table 4).

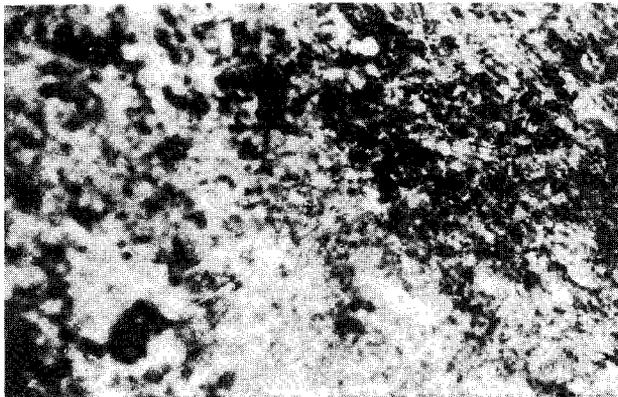
A comparison of all the four test results showed that the influence of the time factor  $tx$  on erosion was much less than that of  $hx$ , irrespective of the ablatively liner used in the combustion chamber. The difference in erosion results between RFM and SPC can be explained as follows.

In the case of RFM, sloughing of chopped silica fabrics occurs during combustion, leading to an increase in the effective shear force of the exhaust gases. This results in greater erosion of the NTI and in deposition of silica.

But in the case of SPC ablatively liner, no such sloughing occurs because the woven silica prepreg fabrics are well embedded in the resin matrix, and only insignificant areas (i.e., edges) are exposed to the hot environment. Silica fabrics in this particular case are perpendicular to the gas flow and form a viscous layer that envelops the charred surface. A small part

**Table 4** Calculated heat-transfer factors and time factors for ground qualification tests with erosion figures

Test no.	Chamber pressure $P_c$ , MPa	Throat diam $D_t$ , mm	$\frac{(P_c)^{0.8}}{(D_t)^{0.1}} = hx$	Time factor, $t_x = t/T$	Ablative liner	%Erosion (profile projection method)
1	1.534	30.78	6.410	0.312	RFM	-1.87
2	1.642	30.76	6.770	0.314	RFM	7.60
3	1.364	30.77	5.838	0.410	SPC	0.00
4	1.621	30.77	6.784	0.400	SPC	1.49

**Fig. 3** Stereomicrograph of throat insert surface with very little silica deposit (mag. X15); liner; SPC.**Table 5** X-ray diffraction results obtained from deposited powders

Serial no.	Bragg angle	Measured $d_{hkl}$	Nearest matching values			
			MoSi <sub>2</sub>	SiC	SiO <sub>2</sub> SiO <sub>2</sub>	Mo
1	8.70	5.9176	—	—	—	—
2	11.98	4.3052	—	—	4.250	—
3	13.30	3.8909	3.91	—	—	—
4	16.50	3.2084	—	—	3.130	—
5	21.78	2.4103	—	—	2.450	—
6	24.23	2.1836	—	2.170	—	—
7	26.63	1.9991	2.03	—	—	—
8	32.07	1.6844	—	—	1.660	—
9	34.90	1.5645	—	—	—	1.570
10	39.00	1.4223	—	—	1.450	—
11	40.60	1.3754	—	—	1.375	—
12	44.33	1.2816	—	—	—	1.281
13	44.75	1.1078	—	—	—	1.114
14	46.00	1.0941	—	1.087	—	—
15	49.60	1.0454	—	—	1.048	—
16	55.15	0.9942	—	—	—	0.995
17	57.45	0.9753	—	0.972	—	—
18	65.40	0.9280	—	—	—	—
19	67.30	0.9198	—	—	—	—
20	68.45	0.9158	—	—	—	—
21	70.75	0.9086	—	—	—	0.908

of the viscous fluid is also deposited on the throat surface (see Fig. 3).

The occasional negative erosions shown in Table 3 clearly indicate that measurements made with only one technique are not always reliable. It is essential to use both the techniques of profile projection and photomicrographs in a complimentary way to arrive at a definite conclusion. Finally, the measurements on the mass difference confirm the nature of erosion (Table 3). For example, even though both the profile projection and the toolmaker's microscope techniques showed negative erosion in test 1, the mass difference clearly showed positive erosion. In both tests 1 and 2, significant weight changes were noticed, even after heavy deposition of silica. Obviously in test 1 the erosion of higher-density molybdenum

more than overcompensated for the deposit of lower-density silica. For large and localized erosion measurements, the toolmaker's microscope method may be adequate.

For the sake of completeness, the chemical compositions of the deposits on the throat inserts were determined by x-ray diffraction techniques (Table 5). The deposits contained mostly mixtures of SiO<sub>2</sub>, SiC, and traces of MoSi<sub>2</sub>.

The formation of silicon carbide was attributed to the chemical reaction between silica and carbonaceous gases generated during degradation of the ablative liner.

### Conclusions

- 1) The degradation of RFM silica-phenolic composites starts at a much lower heat flux level than that for the stacked silica-phenolic composite of identical composition.
- 2) Degradation of the silica-phenolic liner is the prime cause for the erosion of molybdenum-based nozzle throat inserts.
- 3) Combustion chamber pressure has a more pronounced effect on the erosion of the inserts than the time factor (i.e., ratio of cumulative burning time to the total operating time of the motor).
- 4) The stacked silica-phenolic ablative liner with the molybdenum-based composite for the nozzle throat insert is found to yield minimum erosion of around 1.5% for the given motor parameters.

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