

# Ir-Ru/Al<sub>2</sub>O<sub>3</sub> CATALYSTS USED IN SATELLITE PROPULSION

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**Abstract:** Ir/Al<sub>2</sub>O<sub>3</sub>, Ir-Ru/Al<sub>2</sub>O<sub>3</sub> and Ru/Al<sub>2</sub>O<sub>3</sub>, catalysts with total metal contents of 30% were prepared using the methods of incipient wetness and incipient coimpregnation wetness and were tested in a 2N microthruster. Their performances were then compared with that of the Shell 405 commercial catalyst (30% Ir/Al<sub>2</sub>O<sub>3</sub>). Tests were performed in continuous and pulsed regimes, where there are steep temperature and pressure gradients, from ambient values up to 650 °C and 14 bar. Performance stability, thrust produced, temperature and stagnation pressure in the chamber and losses of mass were analyzed and compared to the corresponding parameters in Shell 405 tests. It was observed that the performance of all the above-mentioned catalysts was comparable to that of the commercial one, except for in loss of mass, where the values was higher, which was attributed to the lower mechanical resistance of the support.

**Keywords:** iridium catalyst, ruthenium catalyst, iridium-ruthenium bimetallic catalyst, hydrazine decomposition, spacecraft propulsion, textural properties, TEM, chloride influence.

## INTRODUCTION

The use of hydrazine monopropellant in the propulsion systems of satellites has opened up new horizons in human knowledge, directly affecting the exploration of natural resources in interplanetary space and on earth, weather forecasting, radio and TV communications earth aerial and maritime navigation [Schmidt, 1984]. The Jet Propulsion Laboratory, JPL, was responsible for pioneering research in this field [Altman and Thomas, 1949].

To be considered a good catalyst the product must be able to start the decomposition of cold hydrazine (at temperatures as low as 275K) with a time delay of less than 15 ms. In addition, its performance must remain acceptable during the entire expected life of the satellite.

There are three main phenomena responsible for degradation of a catalyst: mechanical breakup caused by shear external forces and breakup of the grains caused by a fast increase in internal pressure, both of which contribute to the loss of mass of the catalyst, and the decrease in total specific area produced by the elevated temperatures that can be attained in the bed, which result in an obstruction of pores and metal occlusion due to transformations of the structure of the support [Schmidt, 1984].

Several compounds were tested in hydrazine decomposition catalysis. They were considered inadequate for space applications because of their inaptitude to start the reaction at low temperatures (cold start) with acceptable time delays and their poor mechanical resistance to the stresses of the decomposition process. The development of

the Shell 405 (30%Ir/Al<sub>2</sub>O<sub>3</sub>) catalyst by the Shell Company represented a fundamental technological milestone in the use of hydrazine in satellite micropropulsion systems. This catalyst is able to decompose low-temperature (275 K) liquid hydrazine in a very reproducible manner [Armstrong et al., 1978a, 1978b, 1980]. Thanks to this process, hydrazine monopropellant systems became very simple and highly reliable. Presently, the Shell 405 ABSG catalyst allows hundreds of cold starts (293 K) without appreciable mechanical degradation (at least for the first 100 cold starts) over a million hot pulses (373 K) and continuous shots of several hours, when the catalyst bed reaches temperatures as high as 1373 K, all with a time delays of less than 10 ms.

Shell 405 has an elevated metal content of about 30%, which prevents hydrazine from penetrating deep in the catalyst grain, thereby providing good heat dissipation and reducing metal losses to a minimum. Iridium is deposited on a highly crystalline support, which has interlinked micro and macropores [Schmidt, 1984].

Other countries contributed to the research on hydrazine decomposition catalyst technology. This is the case, for instance, in France, where CNESRO III was developed [Pfeffer, 1976; Valentine, 1977; Schmidt, 1984] and Germany, where a catalyst called KC 12 G was produced by Kalichimie [Hartung, 1974; Hartung et al., 1977; Schmidt, 1984].

The use of ruthenium, which is far cheaper than iridium, is a very promising alternative, mainly for small engines delivering a thrust of 100 N or more, as is the case with some launcher roll control devices and apogee satellite motors [Schmidt, 1984], where total burning time reaches a maximum of 5 minutes. In this case a satellite launching uses about 800 g of catalyst. According to the Engelhard Corporation the cost of the iridium metal precursor is around US\$ 27.00/gram, while ruthenium costs about US\$ 3.60/gram.

During the last twenty years there has been little research on new materials to be employed in satellite propulsion. One of the latest developments was a patent registered by Shell [Armstrong et al., 1978b] for a Ir-Ru/Al<sub>2</sub>O<sub>3</sub> catalyst, suggesting that iridium contents must be kept at an atomic concentration between 30% and 80%. Another communication relates the development of a catalyst containing ruthenium only, called Shell X-4, which is reported to have been used with great success in 330 N thrusters [Schmidt, 1984].

The goal of this work is to compare the main results of an evaluation of Ir-Ru/Al<sub>2</sub>O<sub>3</sub> catalysts in a

2 N thruster with the results obtained with 30%Ir/Al<sub>2</sub>O<sub>3</sub> catalyst prepared in the Catalysis Laboratory, at the National Institute for Space Research (INPE) in Cachoeira Paulista, SP, Brazil and those obtained using the Shell 405 commercial catalyst.

## EXPERIMENTAL

### Catalysts Preparation

The alumina used as the catalyst support was prepared at INPE. To have micro and macropores in the final product, a process of controlled generation of porosity by addition of polymer was used, and it yielded the desired bimodal final pore size distribution. The catalyst obtained had the following characteristics:

- 1) Total specific area: 212 m<sup>2</sup>/g;
- 2) Pore volume (humid point): 0.71 cm<sup>3</sup>/g;
- 3) Grain diameter: 0.6 to 0.8 mm;
- 4) Crystallographic phases:  $\gamma$  and  $\eta$ .

The Ir/Al<sub>2</sub>O<sub>3</sub> catalyst was prepared by the method of incipient wetness [Cruz et al., 1989]. To obtain a metal content of 30%, three impregnation steps were necessary. H<sub>2</sub>IrCl<sub>6</sub> acid solution with a free acid concentration of 0.6 N was used as the precursor of the active metal phase. This product was either prepared in the laboratory at INPE or purchased from the Engelhard Corporation, France. Prior to impregnation, the support was oven dried at 393K for 2 hours. After each impregnation solids were dried in vacuum for 1 hour and reduced in H<sub>2</sub> flow, at 673 K for 4 hours.

The Ru/Al<sub>2</sub>O<sub>3</sub> catalyst, containing 30% metal was prepared by the above methodology, using as the precursor a solution of RuCl<sub>3</sub> with a free HCl acid concentration of 0.22N, purchased from the Engelhard Corporation [Soares Neto et al., 1999].

Two different methods were employed in the preparation of Ir-Ru/Al<sub>2</sub>O<sub>3</sub> catalysts [Soares Neto et al., 1993]:

- a) Incipient wetness method: A series of 30% metal content catalysts, with Ir/Ru mass ratios of 2/1, 1/1 and 1/2 were prepared, always with iridium as the first metal to be deposited. The precursor solutions used were H<sub>2</sub>IrCl<sub>6</sub> 0.6N HCl and RuCl<sub>3</sub> 0.22N HCl. Prior to the impregnation, the support was oven dried for 2 hours at 393 K, after which the resulting solids underwent H<sub>2</sub> flow drying for 2 hours followed by reduction at 673 K for 4 hours. The addition of metal in each of the impregnation steps was always kept below 12%.
- b) Coimpregnation method: The same products were obtained, using the same methodology and the same

precursors as above, but in this case both metals were introduced simultaneously in three consecutive impregnations.

### Catalysts Characterization

The following were used to characterize the catalysts:

Gravimetry - for determination of metal weight;

N<sub>2</sub> Adsorption - for determination of specific surface area and specific pore volume ( $d_{\text{poros}} < 30$  nm);

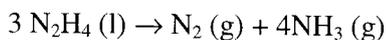
Hg Porosimetry - for determination of specific pore volume ( $d_{\text{poros}} > 30$  nm);

Irreversible H<sub>2</sub> adsorption - for determination of specific number of active sites and dispersion of metal phase, and:

Transmission electronic microscopy (TEM) - for determination of average metal particle size [Soares Neto, 1998].

### Hydrazine Decomposition in a 2N Thruster

The decomposition of N<sub>2</sub>H<sub>4</sub> may occur by the following reactions:



Tests to evaluate the performance of the thruster were conducted in a vacuum chamber simulating space pressure conditions (Figure 1), according to

to the following procedures:

a) Sample drying of the catalyst for about 12 hours at 393 K;

b) Bed packing of the catalyst through the use of a shaking device to reduce voids in the bed, thereby preventing grain vibration during the firings;

c) Assembling of the thruster catalyst bed, using Pt-Rh screens to assure its integrity and to avoid loss of catalyst;

d) Attaching of the nozzle to the set;

d) Installation of the thruster in the vacuum chamber, by linking it to the measurement devices (temperature, pressure, flow rate, and thrust);

e) Establishment of the vacuum inside the chamber and heating of the catalyst bed up to 393 K. Waiting for 2 hours before first firing to remove humidity;

f) Starting of the firing by opening the feed valve, which introduces hydrazine into the bed; and;

g) Monitoring ignition delay, changes in bed temperature (externally), feed line and decomposition chamber pressures, mass flow rate, and thrust.

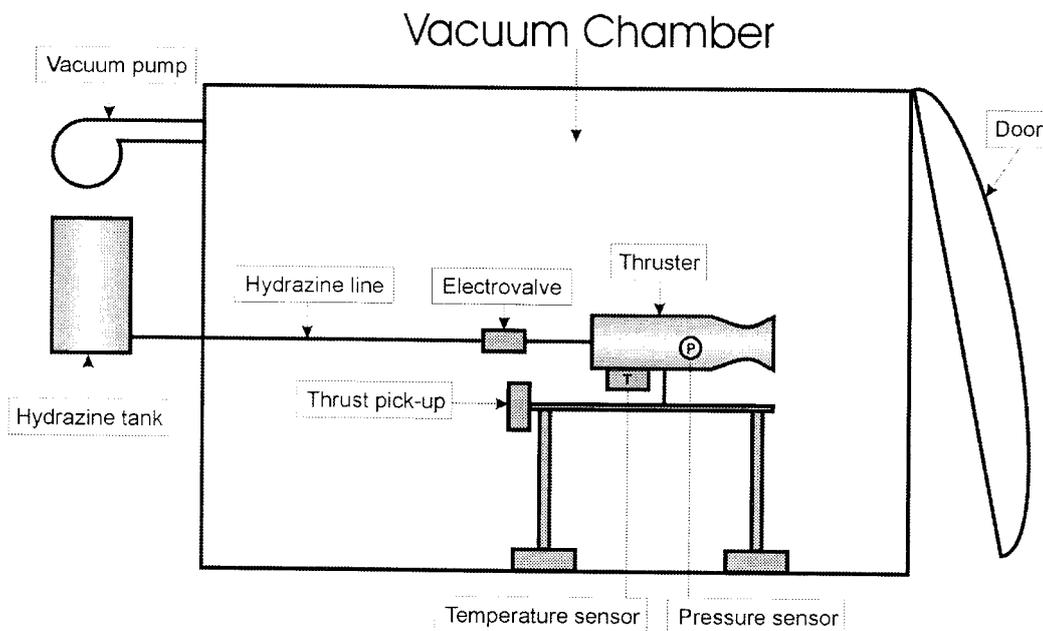
The tests were performed following the same sequence for all catalysts:

a) An initial continuous firing of 200 s;

b) Three firings of 999 pulses, 500 ms each with a 40% duty cycle (feed valve open for 200 ms and closed for 300 ms); and

c) A continuous firing of 200 s.

After the tests, the catalysts were removed from the thruster, dried at 393 K for about 12 hours and weighed to determine the loss of catalysts during the firings.



**Figure 1:** Diagram of the thruster testing apparatus

## RESULTS AND DISCUSSION

Table 1 shows metal contents (on mass and atomic bases) and the Ir/Ru atomic ratio for all catalysts prepared. As the differences between real and nominal metal contents, measured by gravimetric chemical analysis, were below 2%, nominal values shown in Table 1 will be accepted as the real ones.

Table 2 shows the values for specific area, pore volume, number of sites, and dispersion for all catalysts, including Shell 405 which was taken as the reference. It is interesting to note its small pore volume. It can be observed that the addition of 30% iridium barely affects the surface area and specific volume of the alumina, showing that pore occlusion due to introduction of the metal can be neglected. The results obtained by H<sub>2</sub> chemisorption clearly show that ruthenium is much less dispersed than iridium. Even in the bimetallic catalysts, as the ruthenium content increases, the dispersion decreases.

Tables 3 and 4 contain the results obtained by TEM. In Table 3 they show that almost 90% of the monometallic iridium catalyst particle diameters and 57% of the ruthenium ones are shorter than 4.1 nm. The results for bimetallic catalysts fall between these, with ruthenium reflecting the trend towards an increase in the mean size of the metal particles. In Table 4 it can be seen that the mean diameters of ruthenium particles are about 60% higher than those of iridium particles no matter which of the two equations shown is selected for their calculation.

These results also show that the decrease in specific volume of the pores was negligible. This assures that the introduction of 30% metal will not impair the diffusion of gases during the hydrazine decomposition reaction. Despite its introduction, which reduces the available specific surface of the alumina, the metal is still very well distributed in the

final product.

The results of H<sub>2</sub> chemisorption do not agree with those of TEM. In H<sub>2</sub> chemisorption, there is a larger decrease in the dispersion values corresponding to all ruthenium-containing materials than that corresponding to the iridium monometallic catalyst, but this trend was not confirmed by TEM. This could indicate that Ru was not thoroughly reduced by H<sub>2</sub> reduction treatments. This conclusion agrees with the work of Narita et al. [1987a, 1987b].

Line pressure; initial and final temperature of the catalytic bed, as measured on the outer side of the envelope; and stagnation pressure in the chamber are shown in Table 5 for the continuous and pulsed modes. The thrusters usually work in a blow-down regime, which means that the pressure in the tank (and in the line) steadily decreases as pressurizing nitrogen and hydrazine are consumed (Fig. 1). It can also be observed that, as these pressures decrease, the initial and final bed temperatures remain practically unchanged for the Shell 405, but significantly decrease for the other catalysts, in particular in the case of final bed temperature for ruthenium-containing material. In the case of the INPE catalysts, this could be attributable to a certain sensitivity to the decrease in hydrazine flow rate. To examine this hypothesis, tank and line pressures were returned to initial values before each test with the ruthenium catalyst, maintaining hydrazine flow rate and stagnation pressure in the chamber almost constant during the whole series of tests. Even so, the cited temperatures continued to fall.

It can be concluded that the change in hydrazine flow rate is not responsible for the observed temperature decrease. This is caused by the decay of the INPE catalysts as they are used because the rate of loss of mass is still very elevated for these products, in particular for the ruthenium ones, as can be seen in Table 6.

**Table 1: Composition of catalysts**

Sample*	Mass fraction		Atomic fraction		Atomic ratio
	(%) Ir	(%) Ru	(%) Ir	(%) Ru	Ru/Ir
S-Ir	30	0	100	0	0.00
C-2Ir1Ru	20	10	51.3	48.7	0.95
C-1Ir1Ru	15	15	34.5	65.5	1.9
S-1Ir1Ru	15	15	34.5	65.5	1.9
C-1Ir2Ru	10	20	21.8	79.2	3.8
S-1Ir2Ru	10	20	21.8	79.2	3.8
S-Ru	0	30	0	100	-

\* - (C) - Coimpregnation

- (S) - Successive Impregnations

**Table 2: Results of specific area (S<sub>g</sub>), pore volume (V<sub>p</sub>), number of sites, and dispersion.**

Catalyst	S <sub>g</sub> (m <sup>2</sup> .g <sup>-1</sup> ) <sup>(1)</sup>	V <sub>p</sub> (cm <sup>3</sup> .g <sup>-1</sup> ) <sup>(2)</sup>	Sites.g <sup>-1</sup> (x 10 <sup>20</sup> )	Dispersion (%)
Shell 405	114	0.16	4.3	45.8
S-Ir	145	0.41	4.6	49.0
C-2Ir1Ru	134	0.37	1.5	13.0
C-1Ir1Ru	131	0.38	1.3	9.2
S-1Ir1Ru	147	0.39	1.4	10.0
C-1Ir2Ru	134	0.35	1.2	8.0
S-1Ir2Ru	147	0.40	1.3	8.9
S-Ru	128	0.39	1.0	5.6

1 - In 1 g of catalyst, 17 g alumina has a specific surface area of 148 m<sup>2</sup>.

2 - In 1 g of catalyst, 17 g alumina has a specific pore volume of 0.45 cm<sup>3</sup>.

**Table 3: Distribution of the metal particle diameters of the catalysts, obtained by TEM**

Catalyst	n <sup>(1)</sup>	Percentage of particle diameters in each range				
		0 - 2	2.1 - 4	4.1 - 8	8.1 - 12	> 12
S-Ir	1620	41.33	46.66	11.35	0.48	0.18
C-2Ir1Ru	1741	34.24	45.43	19.59	0.51	0.23
C-1Ir1Ru	1636	31.66	45.48	20.97	1.65	0.24
S-1Ir1Ru	1568	42.70	40.08	16.01	1.21	0.00
C-1Ir2Ru	1330	25.92	44.20	27.88	1.51	0.49
S-1Ir2Ru	1622	32.75	44.70	20.83	1.72	0.00
S-Ru	1641	15.23	41.68	36.26	5.86	0.97

1 - Number of particles counted

**Table 4: Mean of the diameters metal particles of the catalysts, obtained by TEM**

Catalysts	$\bar{d}_{p(L)}$ (nm) <sup>(1)</sup>	$\bar{d}_{p(A)}$ (nm) <sup>(2)</sup>
S-Ir	2.4	4.7
C-2Ir1Ru	2.8	5.5
C-1Ir1Ru	2.9	5.4
S-1Ir1Ru	2.7	4.8
C-1Ir2Ru	3.2	6.0
S-1Ir2Ru	3.1	5.2
S-Ru	4.2	6.8

$$1 - \bar{d}_{p(L)} = \frac{\sum n_i \cdot d_i}{\sum n_i}; \quad 2 - \bar{d}_{p(A)} = \frac{\sum n_i \cdot d_i^3}{\sum n_i \cdot d_i^2}$$

**Table 5: Performance of catalysts in 2N microthruster tests**

Catalyst	P <sub>L</sub> <sup>(1)</sup> (bar)	T <sub>i</sub> P <sup>(2)</sup> (°C)	T <sub>f</sub> P <sup>(3)</sup> (°C)	P <sub>c</sub> <sup>(4)</sup> (bar)	Regime <sup>(5)</sup>
Shell 405	21.7	102	830	13.4	Continuous
	20.6	100	830	13.0	Pulsed
	20.0	98	830	12.0	Pulsed
	19.9	96	830	11.9	Pulsed
	19.0	95	830	11.8	Continuous
SIr	21.8	102	770	14.0	Continuous
	22.1	105	770	14.7	Pulsed
	21.5	116	760	14.5	Pulsed
	21.0	100	750	14.0	Pulsed
	20.5	89	730	13.8	Continuous
C2Ir1Ru	20.9	120	750	13.9	Continuous
	20.5	117	715	14.0	Pulsed
	20.0	121	715	13.9	Pulsed
	19.6	117	710	13.4	Pulsed
	19.3	89	695	12.9	Continuous
C1Ir1Ru	21.6	113	730	14.0	Continuous
	20.5	105	695	13.5	Pulsed
	19.5	111	685	13.4	Pulsed
	18.7	114	675	12.8	Pulsed
	19.9	100	675	13.5	Continuous
S1Ir1Ru	21.3	115	735	13.8	Continuous
	20.6	117	680	14.0	Pulsed
	20.8	115	675	13.9	Pulsed
	20.0	116	670	13.6	Pulsed
	19.4	115	670	13.1	Continuous
C1Ir2Ru	21.6	119	710	13.9	Continuous
	20.8	112	680	14.9	Pulsed
	20.0	115	675	13.4	Pulsed
	21.3	114	675	13.9	Pulsed
	20.5	104	675	13.9	Continuous
S1Ir2Ru	21.9	126	730	14.3	Continuous
	21.2	118	660	14.4	Pulsed
	20.6	122	650	14.4	Pulsed
	20.1	121	645	13.9	Pulsed
	19.5	110	635	13.5	Continuous
SRu	21.5	120	735	14.6	Continuous
	21.5	124	710	14.6	Pulsed
	21.5	122	710	14.6	Pulsed
	21.5	123	710	14.6	Pulsed
	21.5	108	645	14.6	Continuous

1 - Hydrazine line pressure; 2 - Initial temperature in the thruster; 3 - Final temperature in the thruster;  
4 - Stagnation pressure in the chamber; 5 - Firing regime

**Table 6: Data on loss of mass**

Catalyst	Initial mass (g)	Loss of mass(%)
SIr	0.61466	11
CIr2Ru1	0.61389	17
CIr1Ru1	0.61056	16
SIr1Ru1	0.63806	18
CIr1Ru2	0.63232	14
SIr1Ru2	0.62241	16
SRu	0.62724	17
Shell 405	1.01772	3.7

Figures 2 to 5 allow a better visualization of the performance of different catalysts in the thruster. One evaluation of these test results showed that, for initial bed temperatures of 373 K, all catalysts had a similar ignition delay of about 20 ms. According to Armstrong et al. [1978b], there are very significant differences in ignition delay between the Ir/Al<sub>2</sub>O<sub>3</sub>, Ru/Al<sub>2</sub>O<sub>3</sub>, and Ir-Ru/Al<sub>2</sub>O<sub>3</sub> catalysts only when decomposition is started at ambient temperature (the so-called "cold starts"). In this case, Ir/Al<sub>2</sub>O<sub>3</sub> has the shortest delay. As the performances of the new catalysts were not known a priori, cold starts were not studied in this work to avoid the possibility of long ignition delays, which could result in hydrazine accumulation in the bed, followed by a hazardous rough initiation, which could damage the thruster and measurement devices.

Figure 2 shows the excellent stability of Shell 405 for all the parameters studied. The slight but steady fall in the pressure curve is due to the classic piezoelectric sensor drift in measurement of constant values: which was not observed in the simultaneous oscilloscope monitoring of this parameter.

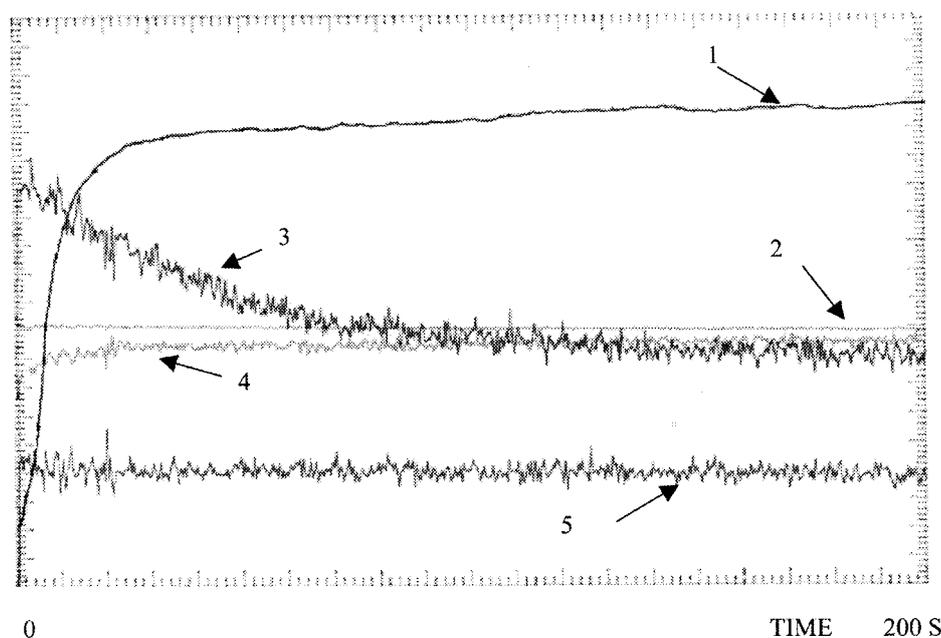
Figure 3 shows that, despite having the same composition, the 30% Ir/Al<sub>2</sub>O<sub>3</sub> catalyst prepared at INPE was slightly less stable than the Shell 405. This demonstrates that the major improvement of the commercial catalyst is because of the properties of the alumina used as the support.

Figure 4 shows the performance of a bimetallic catalyst in pulsed regime. The overpressure peaks observed are probably due to the fact that hydrazine penetrates the catalyst pores and decomposes therein. These peaks should be avoided because they heavily

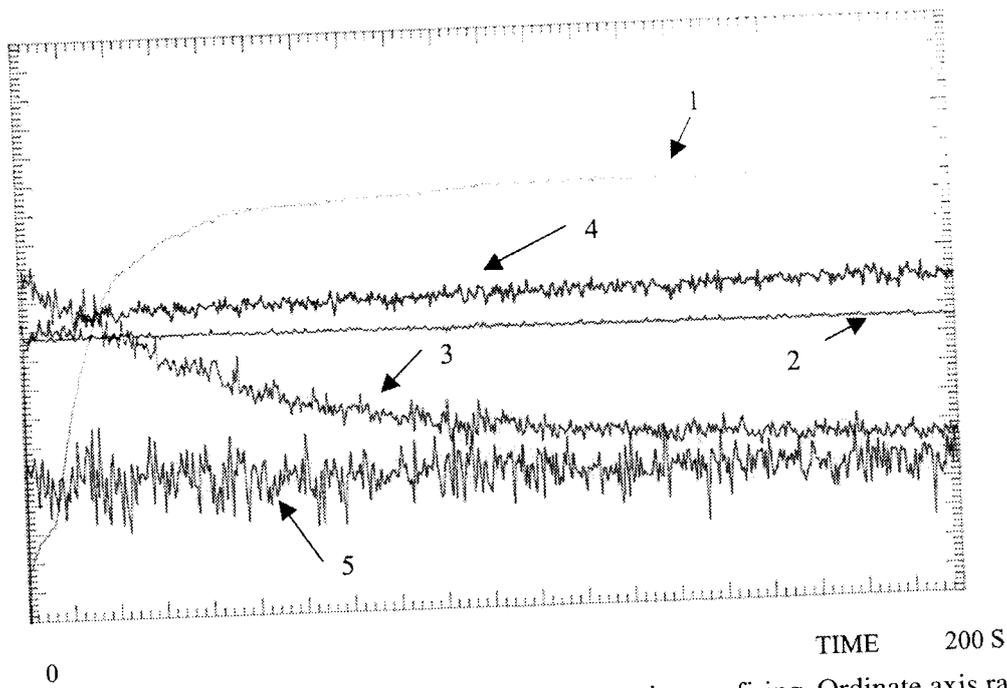
contribute to fractures in the material, causing loss of mass. They were observed more frequently as the ruthenium content increased.

In Figure 5 it can be seen that the ruthenium only catalyst has the highest chamber stagnation pressure instability of all the catalysts. This instability, which has been observed since the first firings with this material, increases with metal content. As the support is always the same, it can be assumed that this is caused by incomplete metal reduction. As a consequence, there is a greater loss of mass when these products are used, which creates more voids in the bed, consequently introducing many preferential paths.

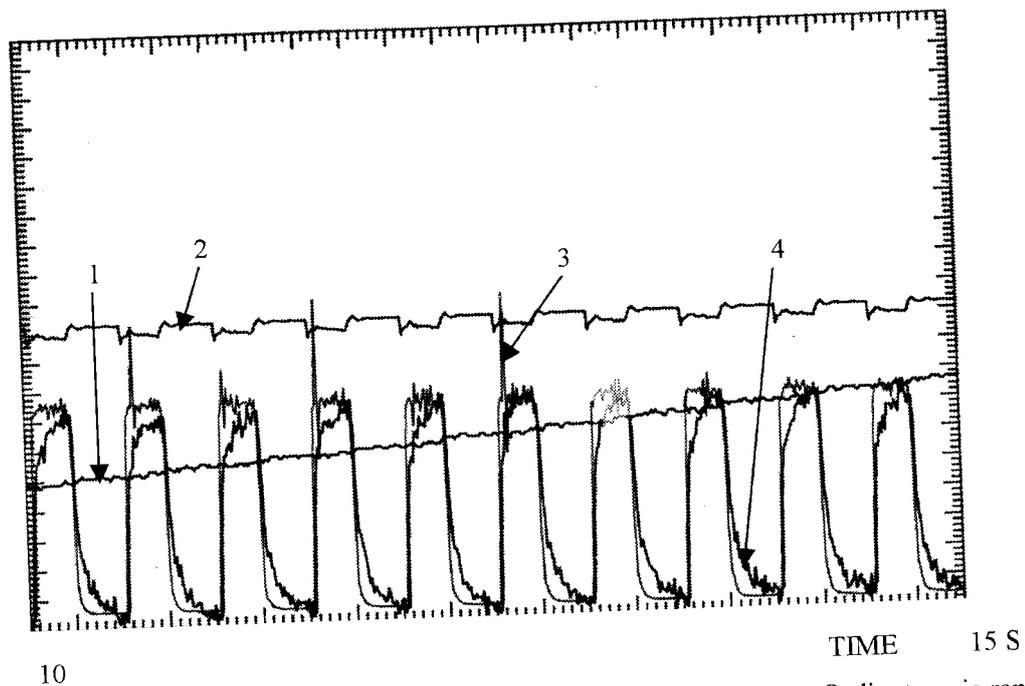
This hypothesis is supported by the data in Table 6, which show the initial mass in the catalytic bed and the corresponding loss mass of during firings. In Table 6 it can be observed that the losses are much higher for the catalysts prepared at INPE than for the Ir/Al<sub>2</sub>O<sub>3</sub> Shell 405 catalyst and that they increase as the ruthenium content increases. This could be associated first with the fact that the alumina prepared at INPE has a lower mechanical resistance and contains a larger amount of macropores and thus yields less dense catalysts. In addition, the incomplete reduction of ruthenium prevents the metal particles from providing an effective protection for the support, which is severely stressed by the thermo-mechanical load associated with heat and mass transfer phenomena during thruster operation. On the other hand, iridium disperses better on the support and has a higher level of reduction, thereby providing better protection to the Shell 405 alumina which, in addition, seems to be intrinsically more resistant than the INPE alumina.



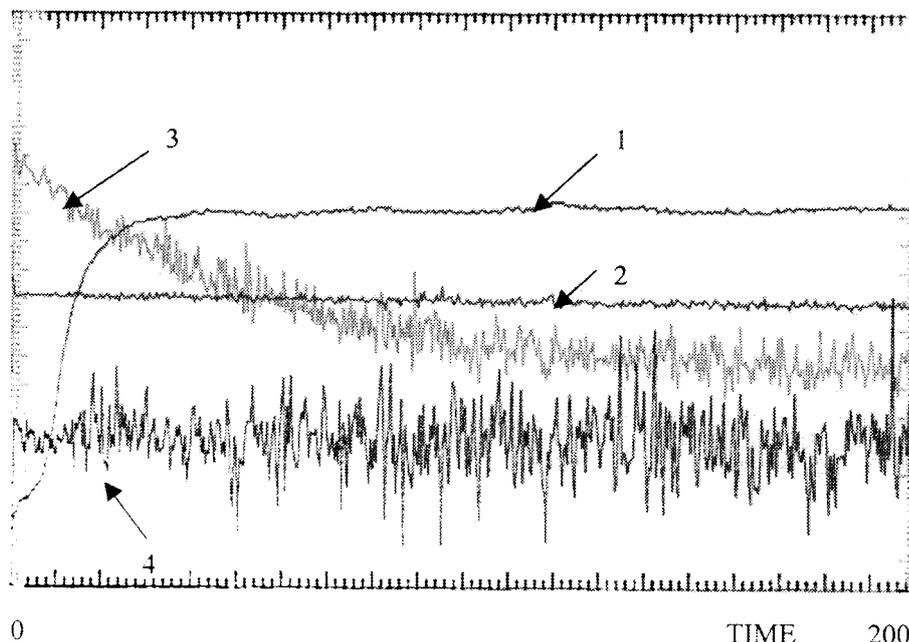
**Figure 2:** Performance of Shell 405 commercial catalyst in the last continuous firing. Ordinate axis range: 1 – Temperature: 0 – 1000 °C; 2 – Line pressure: 0 – 40 bar; 3 – Stagnation pressure in the chamber: –10 – 20 bar; 4 – Thrust: 0 – 4 N; 5 – Flow rate: 0 – 4 g/s



**Figure 3:** Performance of S-Ir catalyst in the last continuous firing. Ordinate axis range:  
 1 – Temperature: 0 – 1000 °C; 2 – Line pressure: 0 – 40 bar;  
 3 – Stagnation pressure in the chamber: – 10 – 30 bar;  
 4 – Thrust: 0 – 4 N; 5 – Flow rate: 0 – 4 g/s



**Figure 4:** Performance of C-1Ir1Ru catalyst in the first pulsed firing. Ordinate axis range:  
 1 – Temperature: 0 – 1000 °C; 2 – Line pressure: 0 – 40 bar;  
 3 – Stagnation pressure in the chamber: – 2 – 38 bar;  
 4 – Thrust: 0 – 4 N



**Figure 5:** Performance of S-Ru catalyst in the last continuous firing. Ordinate axis range:  
 1 – Temperature: 0 – 1000 °C; 2 – Line pressure: 0 – 40 bar;  
 3 – Stagnation pressure in the chamber: – 15 – 25 bar;  
 4 – Flow rate: 0 – 4 g/s

## CONCLUSIONS

The results obtained in this work show that the ruthenium-containing catalysts have problems such as 50% faster mechanical degradation and a higher hydrazine decomposition instability. However, they also demonstrated that the performance of these catalysts was compatible with their use in short-period space applications that require a large amount of catalyst at a cost considerably lower than that of conventional Ir/Al<sub>2</sub>O<sub>3</sub> catalysts. It seems that partial or total substitution of ruthenium for iridium in catalysts for the aforementioned reaction is a very promising procedure. For these new products, it would also be advisable to use alumina with a lower specific volume with the aim of increasing its mechanical resistance.

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