

New Decomposition Catalysts and Characterization Techniques for Rocket-Grade Hydrogen Peroxide

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Traditionally, macroscopic metallic screens and coated ceramic pellets have been used as catalysts for the decomposition of hydrogen peroxide as applied to monopropellant thrusters, liquid rocket engines, and hybrid rocket systems. Catalyst activity depends on available catalytic surface area; metallic catalyst stability is generally degraded by catalyst oxidation. New approaches to forming inexpensive, high surface area decomposition catalyst packs for flightweight applications that do not suffer from oxidative degradation will be discussed. This article will also specifically discuss analytical methods for the assay of hydrogen peroxide and the precise determination of inhibitor species. The synthesis, characterization, and evaluation of high surface area catalyst beds will be discussed as compared to traditional propulsion catalysts. Finally, the effects of valence state and cation type on catalyst activity will be discussed.

Introduction

THE use of hydrogen peroxide as a rocket propellant can be traced back to Helmuth Walter and the Heinkel He 52 aircraft, which was first flown in 1937 (Ref. 1). The 80% hydrogen peroxide was decomposed by parallel feeding liquid catalysts, generally aqueous solutions of calcium permanganate, because of its high solubility in water. The true bipropellant arrived with the Walter 109-509 engine using 30% hydrazine hydrate in methyl alcohol coinjected with 80% hydrogen peroxide.¹

The advent of the first practical heterogeneous catalyst enabled 90% hydrogen peroxide to be used as a monopropellant attitude control thruster on both the X-15 and Mercury spacecraft.² The catalyst used was a pack of silver-plated, 20-mesh brass or nickel screens. The Black Knight program in Great Britain utilized hydrogen peroxide in concert with a silver decomposed through screen pack and a kerosene-based fuel as the first true bipropellant system using hydrogen peroxide as a heterogeneous catalyst pack.²

The U.S. Navy started using 70% hydrogen peroxide in their Mk 16 torpedo in 1955 (Ref. 3). Earlier torpedoes used a homogeneous catalyst of calcium permanganate dissolved in water to react with 48% hydrogen peroxide; the new variant used a heterogeneous catalyst pack to initiate controlled decomposition of 70% hydrogen peroxide prior to mix with the kerosene-based fuel.

The current technology for the production of a heterogeneous catalyst pack is to use silver-electroplated stainless-steel screens, between 40–12 mesh, which are either activated with dilute nitric acid, or promoted via a calcined samarium oxide coating. These screens nominally have a relatively large pressure drop, are relatively dense, and are readily poisoned with the typical contaminants inherent to the use of hydrogen peroxide.

The current research is the development of a low-pressure drop catalyst pack having a high activity and stability, a low weight, and a low cost.

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Analytical

Refractometry

Refractometry allows a quick, precise, and low-volume alternative to concentration determination via hydrometric methods. Approximately 40 mg of sample are required as compared to 110–170 g for a hydrometer. A refractive index determination takes 40 s to perform, which makes it ideal for assay purposes as well as kinetic studies.

A Milton Roy model Abbe-3L refractometer operating at 25°C was used to measure all refractive indices at the sodium D line (589.3 nm). The calibration of the instrument was verified with various immersion oils and a calibration standard supplied by the manufacturer ($n_D = 1.5113$).

Aqueous H_2O_2 solutions were prepared by dilution of 82.1 wt% stock material with doubly distilled water. The H_2O_2 weight fractions of these solutions were determined by potassium permanganate titration techniques. The refractive indices of the above solutions were used to generate Fig. 1. These data are in excellent agreement with values quoted in the literature.⁴

Densitometry

Density is another easily quantifiable physical parameter that varies significantly with H_2O_2 weight fraction. The den-

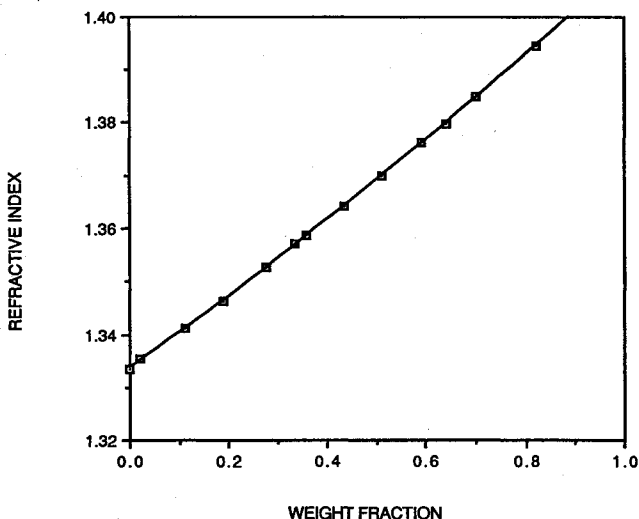


Fig. 1 Refractive index of hydrogen peroxide measured at 25°C as a function of weight fraction.

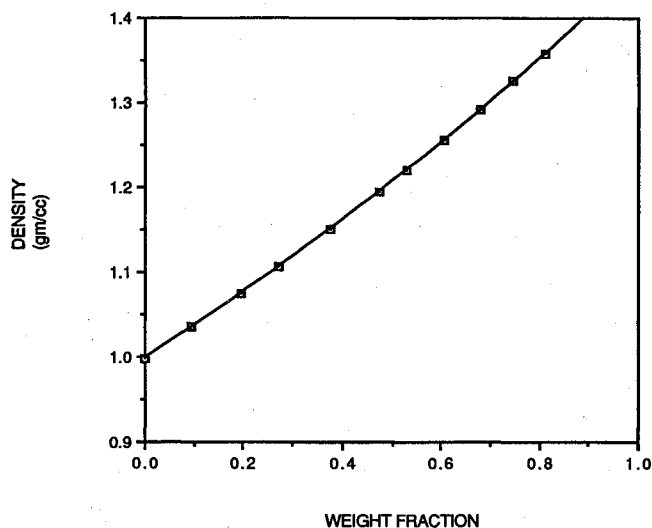


Fig. 2 Density of hydrogen peroxide measured at 25°C as a function of weight fraction.

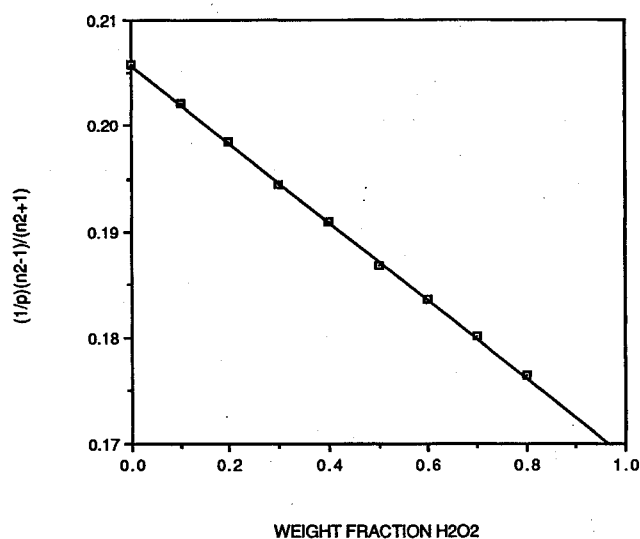


Fig. 3 Graphical representation of the coupling between density and refractive index.

sities of aqueous solutions were measured with an Anton Paar DMA-48 ultrasonic densitometer. Although the required sample volume is approximately 0.7 ml for this instrument, 5 ml was used to flush the oscillator tube to ensure removal of all gas bubbles. Figure 2 shows the measured solution density ρ as a function of H_2O_2 weight fraction at 25°C.

An internal check of the previous refractive index and density measurements is provided by the following relationship from molecular optics⁵:

$$\frac{1}{\rho} \frac{n^2 - 1}{n^2 + 1} = \sum x_a \frac{P_a}{M_a} \quad (1)$$

where x_a is the weight fraction of liquid a of molecular refractivity P_a and molecular weight M_a . In Fig. 3, Eq. (1) is plotted as a function of weight fraction H_2O_2 for the experimentally determined n and ρ .

Spectrometry

Of major importance to the rocket community is the identification and quantification of inhibitors added prior to shipment of the hydrogen peroxide from the manufacturers. Although the precise details of hydrogen peroxide are generally

company proprietary, the industry has essentially used three general modes of inhibition. Lower concentration hydrogen peroxide (15 vol%) uses acetaldehyde as an organic inhibitor. Medium (35 vol%) and high (70 vol%) strength hydrogen peroxide are inhibited by phosphoric acid derivatives as well as sodium stannate. Technical grade hydrogen peroxides generally contain 10–30 mg/l of sodium, tin, and phosphorus. Semiconductor grade material is generally tenfold more clean, 1–3 mg/l, and rocket grade is again generally tenfold more clean, at 0.1–0.3 mg/l.

Sodium, tin, and phosphorus inhibitor concentrations were quantified by using an Applied Research Laboratory model 3410 inductively coupled argon plasma spectrometer (ICP). Calibration curves of line intensity vs element concentration were generated from standard solutions.

Catalyst Synthesis

Catalyst Bases

The catalyst bases used in this study were all comprised of high-surface alumina or silica-alumina-derived compounds. The physical shape of the particles were either oil-dropped/formed spheres or extrudate of a cylindrical geometry. The particle diameters varied from 1/16 to 1/8 in. Table 1 summarizes the catalyst base types and properties as obtained from the manufacturers.

Impregnation

Initial testing of metal oxides was accomplished in concert with a literature review to narrow the field of monometallic catalysts studied. The results of this testing pointed to the oxides of silver, ruthenium, manganese, lead, vanadium, and chromium as target species for further research.

Soluble salts of the previous cations were dissolved in water to obtain solutions at a defined metals concentration of 0.96 mole/l. Silver was obtained from silver nitrate, ruthenium from acidified ruthenium chloride, manganese from both manganous nitrate and potassium permanganate, lead from lead nitrate, vanadium from basic ammonium metavanadate, and chromium from ammonium chromate.

The chosen catalyst base was dried overnight at 300°C to remove volatiles. The weighed catalyst base was then covered with a given catalyst solution and allowed to soak for 4 h. Generally, one-half the volume of catalyst base was used for the volume of impregnation solution. This was adjusted for variances in the catalyst base apparent bulk density. After the 4-h impregnation time, the catalyst was dried in a convection oven at 80°C for 15 h and weighed. The weight change yielded the gravimetric impregnation loading.

Calcination

The phase transition temperatures are available in the literature.⁶ Table 2 outlines the decomposition temperatures of the oxide species of interest.

Calcinations were performed in a furnace by roasting in air above the defined transition temperatures for 15 h. The calcined catalysts were allowed to cool to ambient conditions and were then weighed to yield the final gravimetric metal oxide loading.

Catalyst Characterization

Gravimetry

A relatively precise assessment of what is adsorbed by the catalyst base was obtained by gravimetric analysis. The differential weight obtained through the drying and calcination processes was recorded relative to the original weight of the catalyst base. This difference gave the metal oxide loading, and was recorded in weight percent.

Catalyst bases were subjected to the impregnation, drying, and calcination processes using only distilled water. This gave a true reference for a given catalyst base.

Table 1 Types and properties of selected high-surface catalyst bases

Name	Type	Diameter, in.	Composition, w/w	Surface area, m ² /g	Manufacturer
3A	Extrudate	0.125	Zeolite molecular sieve	900	UOP
4A	Extrudate	0.125	Zeolite molecular sieve	900	UOP
5A	Extrudate	0.125	Zeolite molecular sieve	900	UOP
13X	Sphere	0.125	Zeolite molecular sieve	900	UOP
A305CS/S	Sphere	0.089	Al ₂ O ₃	N/A	LaRoche
A305CS/L	Sphere	0.125	Al ₂ O ₃	N/A	LaRoche
EAB2	Extrudate	0.063	Al ₂ O ₃	204	UOP
SAB2	Sphere	0.063	Al ₂ O ₃	193	UOP
SAB20	Sphere	0.113	Al ₂ O ₃	203	UOP
SAB10	Sphere	0.063	0.369 SiO ₂ /0.631 Al ₂ O ₃	340	UOP
EAB19	Extrudate	0.063	0.419 SiO ₂ /0.590 Al ₂ O ₃	505	UOP
SAB30	Sphere	0.063	0.747 SiO ₂ /0.253 Al ₂ O ₃	341	UOP
SAB35	Sphere	0.063	SiO ₂	118	UOP

Table 2 Transition temperatures and valence states of active catalytic promoters

Species	Valence	Decomposition temperature, °C
Ag ₂ O	+1	230
Ag	0	Stable
MnO ₂	+4	535
Mn ₂ O ₃	+3	1080
Mn ₃ O ₄	+3/+2	Stable
PbO ₂	+4	290
Pb ₂ O ₃	+3	370
Pb ₃ O ₄	+3/+2	500
PbO	+2	Stable
V ₂ O ₅	+5	1750
VO ₂	+4	Stable
CrO ₂	+4	300
Cr ₂ O ₃	+3	Stable

Gravimetry was coupled with x-ray diffraction to yield metal loading, since the oxide composition coupled with oxide loading determines the metal loading through the gravimetric factor.

X-Ray Diffraction

In lieu of absorption measurements, valence information was inferred from x-ray crystallography data gathered on a Scintag Inc. model XDS-2000 powder diffractometer system, utilizing Ni-filtered Cu K_α radiation. The data were peak fitted and compared to known powder diffraction patterns from an on-line Joint Committee on Powder Diffraction Standards (JCPDS) database.

Because of low loading of the catalyst bases, witness samples of each cation were prepared following the same thermal treatment as the catalyst. Each sample was ground to roughly 320 mesh. Long integration times were chosen to facilitate identification of minor phases (less than 0.5% by weight).

Metals Loading by ICP

Weighed samples of the calcined catalyst were digested in a defined volume of either concentrated hydrochloric acid or aqua regia for 24 h at ambient conditions. The concentrate was then diluted to the proper range for ICP analysis. The acids were run for metals by ICP to provide blanks.

Catalyst Evaluation

Batch Activity Testing

The following equation defines the relationship for a first-order reaction:

$$-\ln Ca/Ca_0 = kt \quad (2)$$

where Ca is the batch hydrogen peroxide concentration at time t , in moles per liter; Ca_0 is the initial batch hydrogen peroxide

concentration, in moles per liter; k is the first-order rate constant, in seconds⁻¹; and t is the time, in seconds. By plotting $-\ln Ca/Ca_0$ as a function of time, it is possible to see if a reaction is truly first-order.

The rate constant k has been defined for a homogeneous system. It is preferable to define it on a catalyst atom specific basis. For this study, a catalyst mole basis was chosen. The new catalytic rate constant k' was defined as the experimentally determined rate constant divided by the catalytic metal loading, in moles of active metal.

The batch reactor used was a 50-ml spherical glass flask containing 20 ml of 70% stabilized hydrogen peroxide (FMC/SC grade). The solution was stirred at a defined angular velocity with a magnetic stirrer and a glass-encapsulated thermocouple was inserted into the solution. At time $t = 0$, a precise weight of catalyst was dropped into the hydrogen peroxide solution. Forty- μ l aliquots were withdrawn from the batch at defined time intervals, which were then analyzed by refractive index. A temperature history was also obtained for each experimental run.

By plotting $-\ln Ca/Ca_0$ vs time, and knowing the catalyst molar metal loading k' , the catalytic rate constant was obtained for a variety of catalyst types.

Thermoprobe Testing

The decomposition of hydrogen peroxide is exothermic; 70% hydrogen peroxide gives off 23 kcal/mole as its heat of reaction, hence, a purely isothermal activity test is difficult. A thermoprobe analogy test was developed in which a defined volume (20 ml) of 30% hydrogen peroxide is reacted with a known volume of catalyst (3.2 ml) in a vigorously stirred spherical reactor (100 ml) at initially ambient conditions. The reactor wetted surface to solution volume ratio is therefore constant, and thermal losses can be quantified during reaction.

Thermal history was obtained for each run; peak temperature and time-to-peak temperature were obtained. The time-to-peak temperature as a function of metal loading for a given volume is a direct indicator of catalyst activity.

Results and Discussion

Hydrogen Peroxide Studies

Three commercial grades of hydrogen peroxide were analyzed that are of interest to the rocket community. Table 3 shows peroxide concentration in weight percent via refractive index and inhibitor level via ICP.

All of the grades in Table 3 do not meet the rocket propulsion inhibitor specifications of 0.1–0.3 mg/l. This is indicative of the current state of hydrogen peroxide manufacture; hydrogen peroxide is mainly used in bleaching and wastewater remediation where inhibitor concentration is not critical. It is important to note that the FMC semiconductor grade most closely matches the grade currently used on the Scout rocket.

X-Ray Diffraction Results

The x-ray results of the witness samples are given in Table 4. The thermal decomposition products of potassium permanganate did not match any combination of known compounds listed in the powder diffraction database. However, there was strong evidence that K_2MnO_4 is one phase that is present. Several possibilities have been proposed, but at this time the exact identities of the decomposition products, and therefore the oxidation states of Mn, remain uncertain.^{7,8}

Catalyst Activity Results

Batch Activity Testing

The batch activity test was first run on a 13X spherical molecular sieve loaded with a potassium-promoted manganese oxide. Figure 4 shows a plot of this catalyst system, as related to the form described by Eq. (2).

The first 1000 s of the reaction are linear. Past that point, poisoning, or another secondary effect causes a deviation from linearity. The initial slope, up to 1000 s, is the first-order rate constant k . The rate constant was then normalized to the number of active catalyst metal atoms, as discussed previously, to generate k' . A variety of synthesized catalysts was evaluated by the batch activity test method discussed in the previous section. The results of these activity tests are shown as Table 5.

For a given catalyst base, adsorption varies widely as seen from the impregnated loading values (Table 5, column three).

Table 3 Comparison of various commercial grades of hydrogen peroxide

Concentration	P, mg/l	Na, mg/l	Sn, mg/l	Source
63.9	0.68	2.01	0.67	FMC/SC 70
62.3	15.9	23.8	6.83	SOLVAY 70
80.8	0.75	3.84	1.33	SCOUT 90

Table 4 Summary of XRD witness sample analyses

Starting material	Temperature, °C	Phase
$Mn(NO_3)_2$	210	MnO_2
	450	MnO_2
	600	Mn_2O_3
$KMnO_4$	450	Unknown
	600	Unknown
$Pb(NO_3)_2$	280	PbO
$RuCl_3$	600	RuO_2
NH_4VO_3	210	V_2O_5
	600	V_2O_5

The initial impregnation solutions were all fixed at 0.96 mole/l of active catalytic metal, yet the loaded catalyst values indicate a factor of 180 difference between the smallest loading, derived from ruthenium chloride, to the largest, derived from potassium permanganate. This variance is mostly because of the differing adsorption characteristics of the metal ions with respect to the catalyst bases.

The difference between the impregnated loading value and the calcined loading value indicates the degree of mobility of the metallic precursor/oxide during the calcination process. The most volatile species appears to be manganese derived from manganous nitrate, which shows a loss of a factor of 52 in metal concentration during calcination. Note that there is negligible loss of manganese upon calcination if potassium permanganate is used as the impregnation salt.

Relative activity is defined with respect to the lowest rate constant catalyst studied, in this case, lead oxide. It is interesting to note that all of the manganous species show a similar relative activity of 15, with the exception of the trivalent oxide, Mn_2O_3 , which indicates a slightly lower value. It is also important to note that the addition of a base promoter to manganese apparently does not affect the catalyst relative activity. Ruthenium oxide is seen to have a high relative activity, but the loading is quite low, almost in the noise of the analytical technique, hence, the value should be viewed with caution. A surprising result is seen in the case of silver oxide, among the least active of the catalysts.

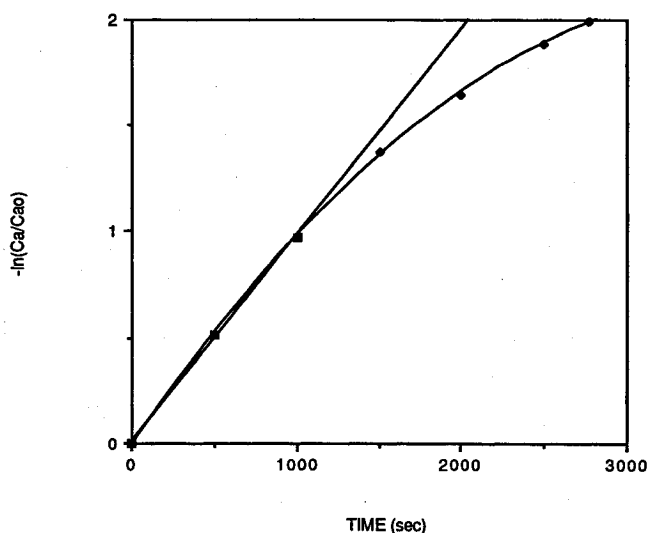


Fig. 4 $-\ln Ca/Ca_0$ as a function of time for the catalyst system K/Mn_xO_y on 13X spherical molecular sieves.

Table 5 Batch activity testing results for selected catalysts and bases using 70% FMC/SC hydrogen peroxide

Base	Catalyst	Impregnated loading, wt%	Calcined loading, wt%	k , $10^{-6} s^{-1}$	k' , $s^{-1} mol^{-1}$	Relative activity
A305CS/L	Ag_2O	1.10	1.18	19.3	0.580	1.5
A305CS/L	RuO_2	0.04	0.01	4.18	15.5	41
A305CS/L	MnO_2	7.27	0.14	19.4	5.67	15
A305CS/L	MnO_2	7.27	0.23	30.4	5.93	16
A305CS/L	Mn_2O_3	7.27	0.33	38.0	4.45	12
A305CS/L	K, Mn_xO_y	0.15	0.09	10.9	5.17	14
A305CS/L	K, Mn_xO_y	0.15	0.16	40.6	5.58	15
A305CS/L	PbO	1.50	1.61	8.35	0.376	1
A305CS/L	V_2O_5	0.42	0.40	8.3	0.485	1.3
EAB2	K, Mn_xO_y	N/A	1.85	133	6.12	16
SAB2	K, Mn_xO_y	N/A	3.68	146	9.69	26
SAB10	K, Mn_xO_y	N/A	1.91	54.9	10.8	29
EAB19	K, Mn_xO_y	N/A	2.61	158	4.07	11
SAB30	K, Mn_xO_y	N/A	2.64	111	9.87	26

Relative activity values are referenced to lead oxide specific rate in all cases.

Table 6 Thermoprobe analogy testing results of heterogeneous propulsion catalysts using 30% reagent grade hydrogen peroxide

Base	Catalyst	Loading, wt%	Total weight, g	ΔT_{MAX} , °C	Time to ΔT_{MAX} , m
13X	K, Mn _x O _y	1.35	1.909	73.3	4.7
SAB10	K, Mn _x O _y	2.63	1.472	61.0	6.3
Al ₂ O ₃	Pt	0.50	2.816	68.6	0.9
PUROFIL	(MnO ₂)	N/A	2.063	56.2	0.6
SS Screen	Ag/SmO ₂	N/A	4.314	54.6	12.9

All tests were conducted at a constant 3.2 ml catalyst volume.

Table 7 Thermoprobe analogy testing-derived parameters and results for supported catalysts in 30% reagent grade hydrogen peroxide

Base	Catalyst	Metals loading, wt%	Catalyst volume, mm ³	r_{eff} , mm	Total batch metals, mmol	Time to ΔT_{MAX} , m	Activity, h ⁻¹
13X	K, Mn _x O _y	1.35	666	5.4	0.0140	30.4	1.97
13X	K, Mn _x O _y	1.35	1330	6.8	0.0286	9.9	6.06
13X	K, Mn _x O _y	1.35	2660	8.6	0.0526	2.5	24.0
Al ₂ O ₃	Pt	0.50	116	3.0	0.00497	9.2	6.52
Al ₂ O ₃	Pt	0.50	231	3.8	0.00902	3.8	15.8
Al ₂ O ₃	Pt	0.50	462	4.8	0.0185	2.0	30.0
A305CS/L	MnO ₂	0.23	293	4.1	0.0144	105.0	0.57
A305CS/L	MnO ₂	0.23	586	5.2	0.0282	64.1	0.94
A305CS/L	MnO ₂	0.23	1170	6.5	0.0601	33.3	1.80
A305CS/L	K, Mn _x O _y	0.09	293	4.1	0.00528	124.2	0.48
A305CS/L	K, Mn _x O _y	0.09	586	5.2	0.0107	56.2	1.07
A305CS/L	K, Mn _x O _y	0.09	1170	6.5	0.0222	38.5	1.56

Activity is defined as the inverse of the induction time.

The effect of varying catalyst bases with a constant impregnation solution is seen as the last five entries. SAB2 adsorbed twice as much active metal catalyst as the EAB2 variant. Even with the low adsorption of SAB10, the catalyst derived from SAB10 proved to have the highest relative activity in this series of runs.

Thermoprobe Analogy Testing

Thermoprobe testing was done on a number of traditional and proposed rocket propulsion catalysts and bases. The results of these tests are shown as Table 6.

PUROFIL is not a true catalyst, but a reactant. It forms manganese dioxide in contact with hydrogen peroxide. The oxide formed has a very high surface-to-volume ratio, as it is in the colloidal state. The main drawbacks of PUROFIL in propulsion applications are the downstream contamination of the rocket combustion region and its quick disappearance during the action time of the rocket; it is included for completeness.

As discussed previously, the time to attain a maximum temperature rise is a direct indicator of catalyst activity. The actual maximum temperature is an indirect measure of activity, folded into heat loss of the batch reactor. It is seen from Table 6 that the most active species is PUROFIL.

Dispersed platinum on alumina is a very active catalyst, followed by the manganese loaded 13X and SAB10 variants. The least active catalyst was the samarium-promoted silver screen, the conventional peroxide decomposition catalyst.

To further understand the effects of equivalent loading, a series of experiments was conducted, varying the number of catalyst pellets for a fixed volume of hydrogen peroxide. Table 7 shows the results of this testing for four catalyst variants.

Effective volume is calculated by measuring the catalyst pellets, calculating a volume per pellet and multiplying by the total number of pellets used in an experiment. The effective radius is the radius derived by assuming a sphere having that effective volume. Total batch metals is obtained by knowing the total catalyst weight, multiplying by the metals loading, and then converting to a molar basis. Activity is defined as the inverse of the time to maximum temperature, for this experimental geometry.

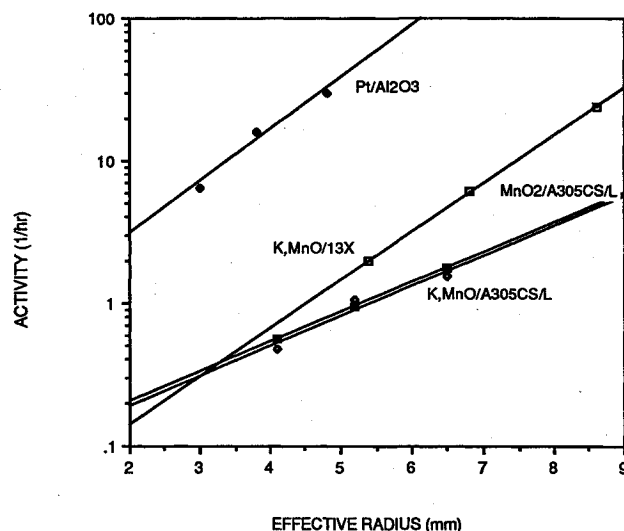


Fig. 5 Log activity as a function of log effective radius derived from thermoprobe analogy tests.

The 13X base with potassium-promoted manganese and Pt on alumina catalysts both have a relatively high surface area. This is directly reflected in their activity values. The A305CS/L base catalysts had considerably lower activity than the previous two.

Data contained in Table 7 have been plotted as Figs. 5 and 6. The log of catalyst activity as a function of the effective radius is shown as Fig. 5. The curves depicted within Fig. 5 are seen to approximate straight lines, within the bounds of the experiments. If anything, the supported Pt catalyst exhibits a slight concavity to the right. This implies that the supported Pt catalyst will saturate in activity with increasing effective radius.

Figure 6 illustrates the log of catalyst activity as a function of the log of total molar batch metals concentration. The rate of change of activity with concentration is most noticeable with the 13X catalyst. The overall catalyst activity is the same at a loading of roughly 0.02 mmol/batch. Above this concen-

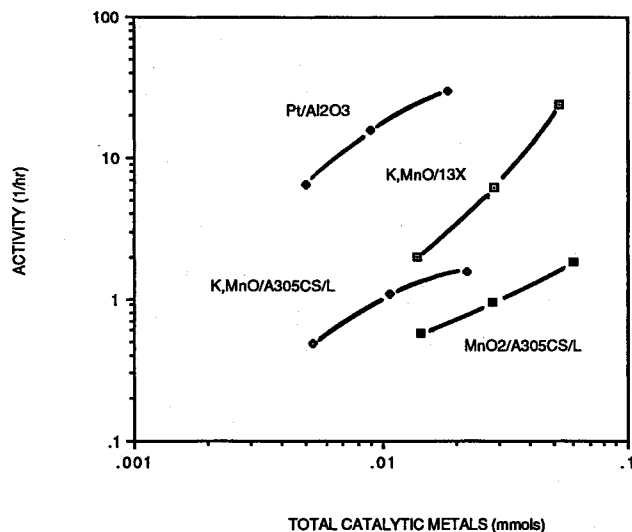


Fig. 6 Log activity as a function of log batch metals concentration derived from thermoprobe analogy tests.

tration, the manganese oxide is a better catalyst, on a high-surface substrate; below this value, the platinum is superior. The method to enable this within a given catalyst volume is to increase the metals loading of the catalyst particles.

All of the activity testing was done in the batch mode to assess catalyst activity. Another important parameter is catalyst stability, especially in a rocket motor of relatively long duration, subject to throttling, or shutdown and startup cycles. To fully address catalyst stability for these applications, flow systems through a fixed catalyst bed with proper instrumentation are desired, which will be the focus of future work.

Conclusions

The following conclusions are obtained from the batch activity testing of hydrogen peroxide decomposition by heterogeneous catalysts.

1) Low-cost, low-weight catalysts have been formulated with high decomposition activity relative to platinized catalysts.

2) Refractive index is a convenient, low volume, and accurate method to determine the concentration of hydrogen peroxide.

3) High-surface catalyst bases are essential to producing highly active catalysts.

4) Tetravalent manganese is more active than trivalent manganese as a catalytic agent.

5) Base-promoted manganese exhibits lower weight loss during calcination than manganese alone.

6) It appears that neutral silver is more catalytically active than silver oxide.

It is recommended that a flow reactor be used to assess catalyst stability and erosion rates at rocket firing conditions; this work is planned for the near future.

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