TECHNICAL REPORT RK-76-11

SOLID PROPELLANTS FOR GENERATING HYDROGEN

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20 April 1976

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER: RK-76-11	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle): Solid Propellants for Generating Hydrogen		5. TYPE OF REPORT & PERIOD COVERED: Technical Report
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Dr. Orval E. Ayers, Dr. James Murfree, Dr. Pasquale Martignoni, and Dr. William M. Chew		8. CONTRACT OR GRANT NUMBER(s): DA 1X36333114D093, AMCMS 633314.12.26000
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Commander, US Army Missile Command, ATTN: DRSMI-RK, Redstone Arsenal, Ala. 35809		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS: Commander, US Army Missile Command, ATTN: DRSMI-RPR, Redstone Arsenal, Ala. 35809		12. REPORT DATE: 20 April 76
		13. NUMBER OF PAGES: 17
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report): Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

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DA-1-X-36333114-D-093

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Solid propellants
Deuterated compounds
Propellant grains
High purity hydrogen

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Solid propellants have been developed and evaluated in a hydrogen gas generator as potential sources of hydrogen for the hydrogen flouride/deuterium flouride chemical laser. Formulations based on the reaction of sodium borohydride ($NaBH_4$) with ferric acid (Fe_2O_3) to produce high purity hydrogen and the reaction of $NaBH_4$ with $(NH_4)_2SO_4$ to produce a mixture of hydrogen and nitrogen have been investigated. When deuterated compounds are used in place of the hydrogen-containing compounds, deuterium (D_2)

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is generated in place of hydrogen (H_2). Propellant grains of $NaBH_4/Fe_2O_3$ weighing approximately 0.08 kg have been prepared and used to generate high purity hydrogen gas for use as a laser cavity fuel and precombustor fuel in a small hydrogen flouride/deuterium flouride chemical laser device. The propellant formulations that contain $NaBH_4$ are very stable to moisture, exhibit no sensitivity to impact or friction, and have autoignition temperatures above 773 K. These propellants or similar type solid propellants are potential storable H_2/D_2 gas sources that combine instant readiness with excellent handling characteristics and compact storage. The volumetric efficiency of these solid propellants is equivalent to or greater than that of liquid hydrogen as a source of gaseous hydrogen. Experimental data from the hydrogen flouride/deuterium flouride chemical laser tests confirm that hydrogen produced from a solid propellant grain is equivalent to commercial hydrogen for use as a cavity or precombustor fuel in the hydrogen flouride/deuterium flouride chemical laser.

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I. INTRODUCTION

In the past few years, considerable effort has been expended to develop hydrogen fluoride/deuterium fluoride (HF/DF) chemical lasers. These lasers depend on the use of high pressure gaseous or cryogenic liquid reactants and diluents. The replacement of cryogenic or high pressure gaseous materials by storable reactant and diluent sources in the condensed phase is highly desirable for Army field use of the chemical laser.

Thus, long-term storable reactants are needed for advanced chemical laser systems for improvement of system handling, packaging, and safety. Preliminary efforts have been initiated to verify the feasibility of solid storable hydrogen sources for use in HF/DF chemical lasers.[1] The development of storable sources of hydrogen for high energy chemical laser applications was the objective of this investigation.

II. BACKGROUND

Storable sources of hydrogen and deuterium, in a compact form, are needed for the HF/DF chemical laser system. The system must be storable in a standby operational mode for long periods of time under the environmental conditions required by the Army. Storage life and environmental conditions should be similar to those required of missile propellants.[1] An important consideration in the future application of HF/DF chemical lasers is the safety associated with the use of various reactants. Storage, transportation, and handling procedures prior to use and the hazards associated with gas generation as part of the laser operation are aspects of particular importance to the Army where the laser operation is in close proximity to operating personnel.

Both storability and safety restrict the use of cryogenic liquid hydrogen and deuterium in an operational chemical laser. Also, safety and weight considerations make high pressure bottled gas sources appear unattractive for application in Army chemical laser systems. Since there is no significant difference in chemical properties between hydrogen and deuterium gas generators, these H_2/D_2 combinations will be referred to as hydrogen throughout this report. The primary objective of the work described in this report is to avoid the storage and handling hazards associated with high pressure or liquid hydrogen by developing an all-solid propellant composition which, upon ignition, can supply the hydrogen gas for the laser. These solid propellant gas generators will provide a completely storable hydrogen gas source that combines instant readiness with excellent handling behavior and compact storage.

Solid propellant formulations have been developed that generate high purity hydrogen via a thermochemical reaction between a metallic hydride and a relatively nonenergetic oxidizer. Examples of hydrides used are lithium aluminum hydride ($LiAlH_4$), sodium borohydride ($NaBH_4$), sodium aluminum hydride ($NaAlH_4$), lithium borohydride ($LiBH_4$), and aluminum hydride (AlH_3). Ferric oxide (Fe_2O_3) and aluminum oxide (Al_2O_3) are examples of the oxidizer ingredient. The thermochemical reaction produces a solid clinker-like residue and essentially particulate free gas with a purity of 39 mole percent or higher. By using different combinations of hydride and oxide, it is possible to achieve calculated reaction temperatures in the range of 773°K to approximately 3250°K.

The major part of the laboratory effort described in this technical report was on the development and evaluation of a solid propellant based on a $NaBH_4/Fe_2O_3$ formulation.

III. EXPERIMENTAL

A solid propellant has been developed and evaluated as a hydrogen gas generator for use in the HF/DF chemical laser. This generator is based on the reaction of $NaBH_4$ with Fe_2O_3 to produce hydrogen gas according to the following equation:

$$3NaBH_4 + 2Fe_2O_3 \rightarrow 3NaBO_2 + 4Fe + 6\,H_2 \quad . \qquad (1)$$

The thermochemical reaction takes place at a 3/2 molar ratio or a weight ratio of 26/74. Sodium borodeuteride ($NaBD_4$) can be used in place of the $NaBH_4$ in Equation (1) to give a solid composition that generates D_2. A weight ratio of $NaBD_4/Fe_2O_3$ of 28.2/71.8 is used in the deuterium formulation.

Solid propellant grains were prepared by the following general procedure: Moisture free Fe_2O_3 was obtained by drying the material in a furnace at 823 to 873°K for 4 hr to remove any adsorbed moisture or waters of hydration. Stoichiometric ratios of $NaBH_4$ and dry Fe_2O_3 were uniformly mixed in a blending mill or by shaking in a closed container, taking precautions to minimize exposure to moisture. The mixed propellant was then pressed into pellets or metal canisters using a remote hydraulic press at a total load of 907.2 kg (2,000 lb) for approximately 5 min.

Pellets and canisters can be made in various diameters and lengths to produce small or large volumes of hydrogen gas depending on the demand. The rate of hydrogen generation may be varied by varying the diameter of the grain. Grains of the $NaBH_4/Fe_2O_3$ formulation have been pressed into canisters with a diameter of 25.4 mm and up to 101.6 mm in length. Propellants weighing up to 0.08 kg have been prepared by the previously mentioned procedure and evaluated in an operating HF/DF chemical laser.

Grains pressed at 907.2-kg total load had a burning rate of 4.83 mm/sec and 4.06 mm/sec under an inert nitrogen pressure of 3.45 MPa (500 psi) and 13.8 MPa (2000 psi), respectively. The decrease in burning rate of solid hydrogen gas generator propellants at high pressures has been observed by other investigators.[1] Figure 1 shows a plot of burning rate versus pressure for the $NaBH_4/Fe_2O_3$ propellant. Density measurements were made on the solid propellant by pressing small pellets at 907.2-kg total load, weighing, and determining the volume by displacement of an inert liquid such as n-heptane. The theoretical density of the stoichiometric formulation based on the densities of the ingredients is 2.61 Mg/m^3 (g/cc) as compared with an experimental value of 2.50 Mg/m^3 (g/cc).

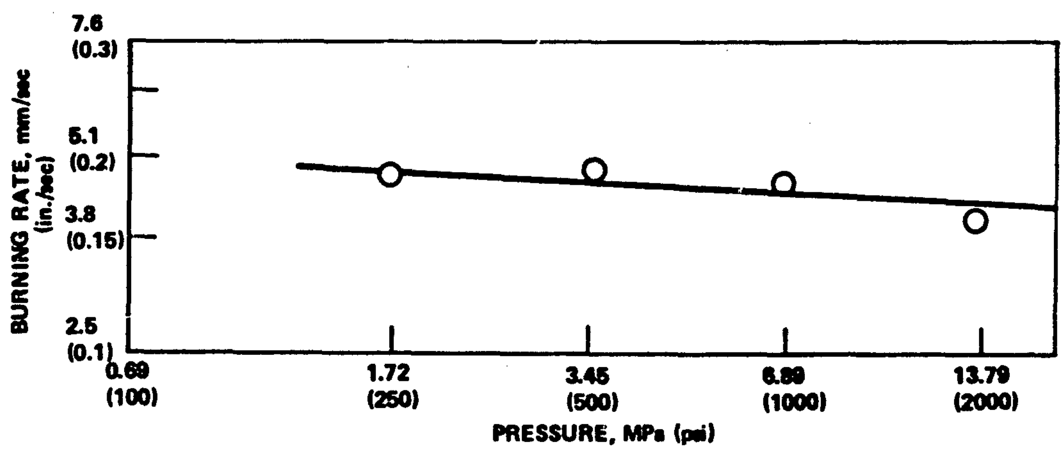

Figure 1. Burning rate versus pressure for $NaBH_4/Fe_2O_3$ propellant.

[1] Dengel, O.H., Naval Surface Weapons Center, White Oak, Maryland, "Private Communication."

Initial laboratory formulation and burning studies were conducted on the propellant using a modified window bomb which was replaced with the combustor described in a recent technical report.[2] Ignition of propellant grains was by use of a hot nichrome wire. Ten volts at approximately 10 amperes were required to produce enough heat in the nichrome wire to initiate the self-sustaining thermochemical reaction in the solid propellant. The reaction went easily to completion and produced hydrogen gas with a purity greater than 99 mole percent as indicated by mass spectrometric analysis. A solid "clinker" residue remained after combustion and gas production.

The solid grain hydrogen generator based on $NaBH_4/Fe_2O_3$ was evaluated in a small scale HF/DF chemical laser device as a laser cavity fuel and as a combustor fuel and the results were compared to those obtained from bottled hydrogen gas[3]. A schematic of the apparatus used for evaluating the solid propellant on a chemical laser device is shown in Figure 2. It consisted of a solid grain combustor with an accumulator tank and a HF/DF chemical laser device consisting of a precombustor injector, variable length precombustor, secondary nozzle, laser cavity and associated feed systems, and exhaust system. A cross-sectional drawing of the secondary nozzle is shown in Figure 3. This is a parallel flow slit nozzle in which fluorine atoms from the precombustor are flowed through the center of the nozzle and mixed with deuterium or hydrogen from the top and bottom slits in the laser cavity. The nozzle is 9.5-mm high by 100-mm long and is water cooled.

Calcium fluoride windows were used on each side of the laser cavity to permit power extraction. These windows were mounted at Brewster's angle on adapter plates with 50.8-mm diameter openings in conjunction with external mirrors. The optical cavity was plano-concave, consisting of a 5-m radius of curvature gold coated mirror and a plane, partially transmitting germanium output mirror coated for 5% transmittance. Power output was measured on a 0- to 100-W power meter.

Cylindrical grains measuring 25.4 mm in diameter and up to 101.6-mm long have been fired in the apparatus using a hot nichrome wire to initiate combustion. The hydrogen gas generator was used to pressurize an evacuated

[2] Chew, W. M., Ayers, O. E., et al., Experimental Combustor for Solid Gas Generator, US Army Missile Command, Redstone Arsenal, Alabama, 1975, Report No. RK-TR-76-2.

[3] Ayers, O. E., et al., "Solid Hydrogen Gas Generator for Chemical Lasers," Bulletin of First DOD Conference on High Energy Laser Technology, 1974.

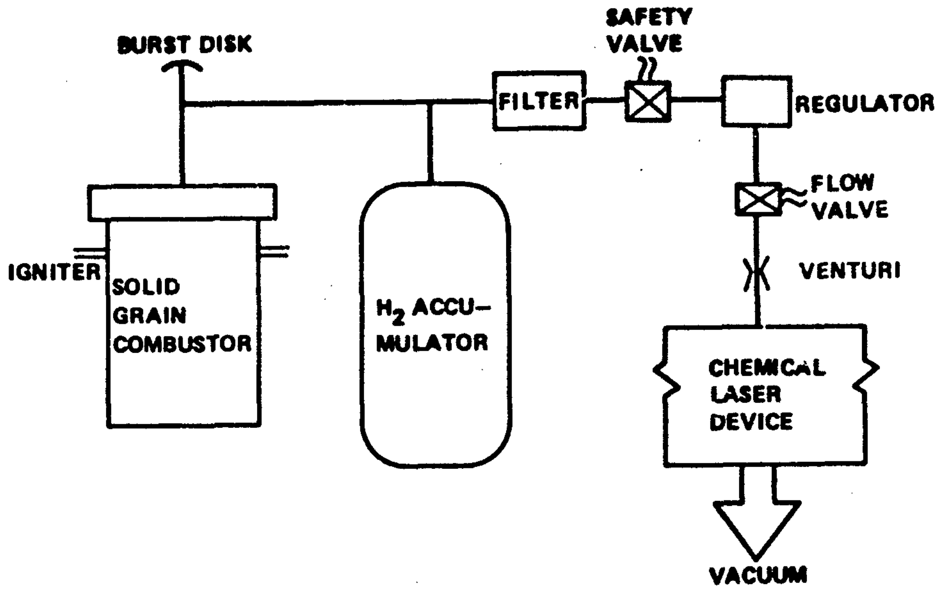

Figure 2. Apparatus for firing solid propellant grain as hydrogen generator for chemical laser.

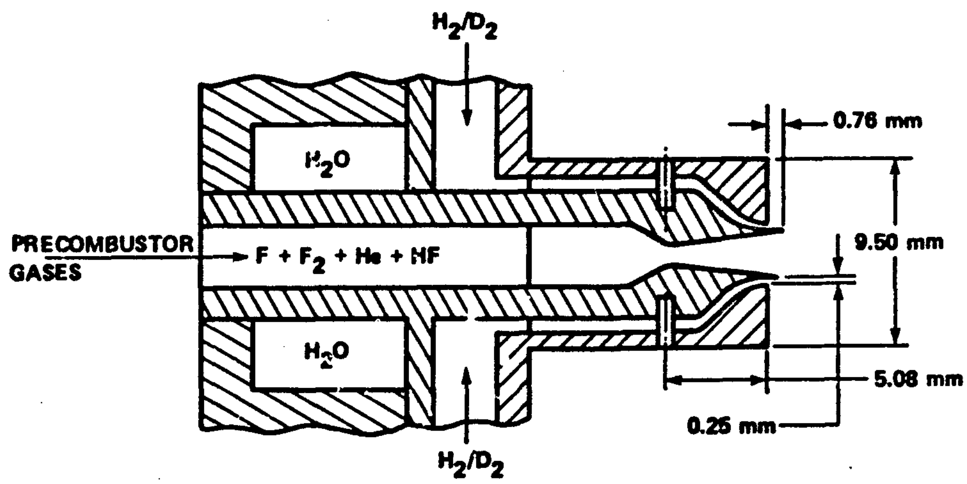

Figure 3. Parallel flow slit nozzle (scale 5/1).

1000-cm^3 accumulator tank with H_2 to approximately 2.45 MPa (350 psi). This hydrogen was then supplied to the chemical laser device using a pressure regulator and cavitating venturi flow control to obtain the data reported in Table 1.

TABLE 1. SUMMARY OF LASER RUNS

	Flow rates (cm^3/sec)						
Laser Species	F_2	Cylinder H_2	Generator H_2	D_2	He	Total	Power (W)
DF	379	202	—	566	745	1892	50.9
	380	202	—	571	734	1887	43.5
	380	202	—	571	734	1887	45.9
	379	—	190	566	739	1874	46.4
	380	—	202	566	739	1887	45.9
	380	—	202	566	739	1887	46.6
	380	—	202	566	734	1882	45.9
HF	380	594	—	196	728	1898	6.0
	381	594	—	196	756	1927	5.0
	381	594	—	196	756	1927	5.9
	381	—	661	196	750	1988	5.5
	381	—	683	196	750	2010	6.0

IV. RESULTS AND DISCUSSION

The major effort of this investigation has been concentrated on solid propellant compositions containing $NaBH_4$. Experimental studies with this metallic hydride have confirmed the feasibility of this approach to a solid hydrogen gas generator. Several solid formulations have been prepared that show reliable ignition and combustion behavior with good retention of the solid products and production of gas free of particulate matter. Hydrogen gas produced by the most promising $NaBH_4/Fe_2O_3$ composition showed gas purity equivalent to or exceeding that of commercial tank hydrogen. This gas generator formulation containing $NaBH_4/Fe_2O_3$ in a 26/74 weight ratio was found to be very stable with no sensitivity to impact or friction and to have a high autoignition temperature. In addition, $NaBH_4$, in contrast to many other hydrides, can be handled in the open in the presence of atmospheric moisture for a limited time and requires no special handling procedures.

A limited amount of experimental data have been obtained on solid compositions containing $LiAlH_4$. This hydride has a low autoignition temperature (423°K) and exhibits a greater sensitivity to impact and friction than $NaBH_4$ in solid propellants. Also, $LiAlH_4$ reacts violently with water and formulations containing this material have to be processed and stored under dry conditions to avoid accidental ignition or deterioration of the solid grain. Other hydrides such as AlH_3, $LiBH_4$, and $NaAlH_4$ have been briefly investigated in solid propellant compositions.

In solid hydrogen gas generators based on formulations such as $NaBH_4/Fe_2O_3$ or $LiAlH_4/NH_4Cl/Fe_2O_3$, the weight of available hydrogen is a small percentage of the total weight of the formulation. In the $NaBH_4/Fe_2O_3$ propellant, approximately 3 weight percent hydrogen gas is produced with a purity of 99 mole percent or better. However, this gas generator gives a favorable volumetric efficiency that is equivalent to that of liquid hydrogen. For example, 1 cm^3 of the solid propellant will theoretically produce 804 cm^3 of hydrogen gas at standard temperature and pressure (STP) as compared to 1 cm^3 of liquid hydrogen producing 778 cm^3 of hydrogen at STP. Samples of the $NaBH_4/Fe_2O_3$ propellant have been fired that produced experimental volumetric efficiencies equivalent to that of liquid hydrogen. Table 2 summarizes the theoretical volumetric efficiencies of several solid propellants containing various metal hydrides. From Table 2, it may be seen that compositions 2 through 4 have higher volumetric efficiencies for H_2 release than the $NaBH_4/Fe_2O_3$ formulation. However, composition 2 with $LiBH_4$ and composition 4 with $LiAlH_4$ are more sensitive to moisture, friction, impact, and heat than composition 1. Thus, the ease of handling of $NaBH_4$ and the $NaBH_4/Fe_2O_3$ formulation compensates for its less efficient production of H_2.

TABLE 2. COMPARISON OF EFFICIENCY OF HYDROGEN GAS GENERATORS

	Composition 1	Composition 2	Composition 3	Composition 4	Liquid H_2
Ingredients	$NaBH_4/Fe_2O_3$	$LiBH_4/Fe_2O_3$	$NaBH_4/(NH_4)_2SO_4$	$LiAlH_4/NH_4Cl/Fe_2O_3$	H_2
Composition (wt%)	26/74	17/83	53.4/46.6	61.2/28.8/10.0	100
Density (Mg/m^3)	2.61	2.41	1.32	1.14	0.07
H_2 at STP/cm^3 Propellant	804	842	1247	940	778
Reaction Temperature (°K)	918	> 513	923	> 423	21 (b.p.)
H_2 Yield (wt%)	2.8	3.15	8.5	7.59	100

The $NaBH_4/(NH_4)_2SO_4$ composition (composition 3 in Table 2) appears to be one of the more promising solid hydrogen gas generator formulations from volumetric and weight yield considerations. An optimum $NaBH_4/(NH_4)_2SO_4$ formulation should produce mainly hydrogen with a small amount of nitrogen which can serve as the inert diluent in the HF/DF chemical laser. Preliminary experimental work on the $NaBH_4/(NH_4)_2SO_4$ composition shows that a gas mixture containing approximately 90% H_2, 4% N_2, 5% NH_3, and traces of other gaseous products is produced from grain combustion.

Tests have been performed on a small screening HF/DF chemical laser to determine the effects of NH_3 in D_2 on the DF laser. As shown in Figure 4, 2% NH_3 in the D_2 will decrease the power output to approximately one-half its original value when compared to pure D_2 as the cavity fuel. Therefore, the production of NH_3 by the $NaBH_4/(NH_4)_2SO_4$ formulation is undesirable. Some of the NH_3 formation may occur during the slow thermal decomposition of the grain as a result of the ignition delay encountered when using the nichrome wire to ignite the solid grain. Additional work is being performed on the $NaBH_4/(NH_4)_2SO_4$ composition to improve the experimental hydrogen yield and eliminate the NH_3 from the gaseous products. Part of this work will be on methods to reduce the ignition delay of pressed pellets of the formulation.

Composition 4, which contains $LiAlH_4$ and NH_4Cl, has been investigated as a potential hydrogen gas generator by the Air Force[4]. This composition produces a larger volume of H_2 per unit weight of formulation and a higher weight percent yield of hydrogen than compositions 1 and 2. However, the composition containing $LiAlH_4$ is more hazardous to handle and process than the borohydride compositions due to the fact that $LiAlH_4$ is more sensitive to impact, friction, heat, and moisture. Also, the $LiAlH_4$ composition leaves a reactive "clinker" residue containing LiH after combustion. Tests have shown that this solid clinker material will spontaneously ignite upon contact with water.

A limited amount of work has been done on the production of high temperature hydrogen gas from solid propellants. A solid propellant formulation containing AlH_3 and Fe_2O_3 has been prepared and combusted to produce hydrogen

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[4] O'Pray, J.E., Air Force Weapons Laboratory, Kirtland, AFB, New Mexico, "Private Communication."

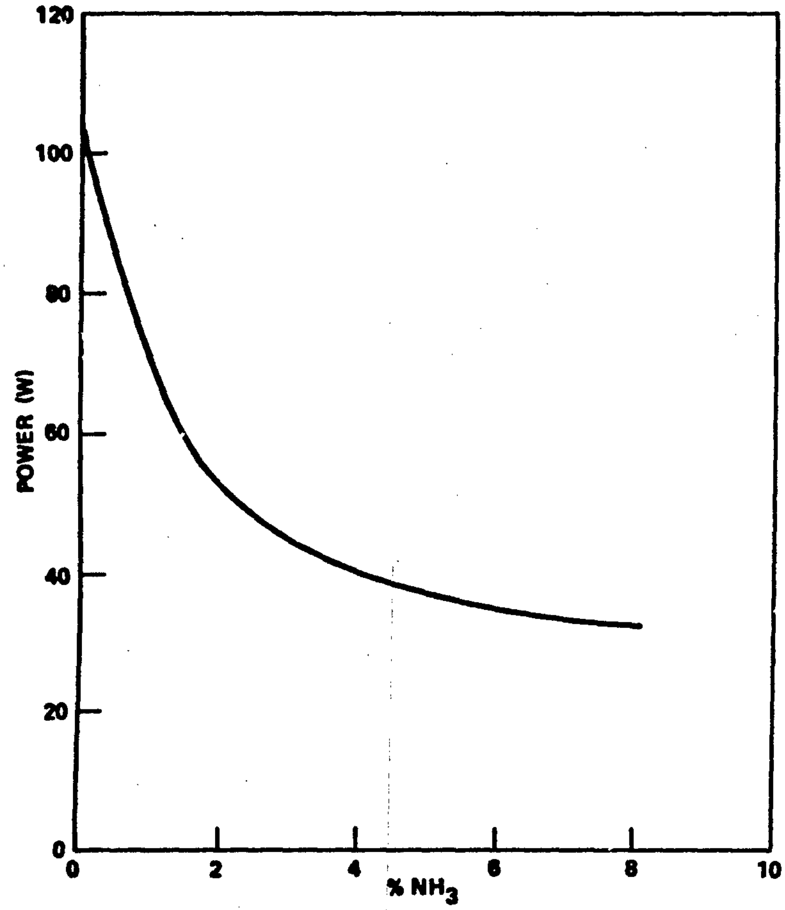

Figure 4. Effects of NH_3 in D_2 on DF laser.

gas at a very high reaction temperature. This involves the reaction of AlH_3 with Fe_2O_3 to produce iron and aluminum oxide with the release of hydrogen gas and a large amount of heat according to Equation (2):

$$2\ AlH_3 + Fe_2O_3 \rightarrow 2\ Fe + Al_2O_3 + 3\ H_2 \quad . \tag{2}$$

The highly exothermic reaction is self-sustaining after initiation and is analogous to the classical thermite reaction. A temperature measurement was not obtained on the reaction, but was estimated to be in the vicinity of the thermite reaction temperature (3250°K).

The solid grain hydrogen generator based on $NaBH_4/Fe_2O_3$, composition 1 in Table 2, was evaluated in a small scale HF/DF chemical laser device as a laser cavity fuel and as a precombustor fuel. The initial lasing runs were made in both DF and HF laser output modes using commercial bottled hydrogen and deuterium to obtain baseline data. These data were compared with those obtained using the solid propellant gas generator as the hydrogen source. All the runs were at a total flow rate ranging from 1874 to 2010 cm^3/sec or approximately 0.001 kg/sec. Each experimental run lasted for a total duration of 10 sec. Hydrogen produced by the solid gas generator was run as a precombustor fuel in the DF laser and as the laser cavity fuel in the HF laser. The following equations show the reactions that are taking place in the HF/DF chemical laser using H_2/D_2 and F_2 as the reactants:

<u>DF Laser</u>

Precombustor: $H_2 + \text{excess } F_2 \rightarrow HF + F_2 + F$

Laser Cavity: $D_2 + F \rightarrow D + DF^*$

$D + F_2 \rightarrow F + DF^*$

<u>HF Laser</u>

Precombustor: $D_2 + \text{excess } F_2 \rightarrow DF + F_2 + F$

Laser Cavity: $H_2 + F \rightarrow H + HF^*$

$H + F_2 \rightarrow F + HF^*$.

The experimental data obtained in the evaluation of the solid hydrogen gas generator on the small HF/DF chemical laser are shown in Table 1. The various experimental runs give a direct comparison of bottled hydrogen with the hydrogen produced by the solid propellant gas generator. In the DF laser, both bottled hydrogen and H_2 from the solid gas generator were run as precombustor fuels. Both sources of hydrogen gave approximately the same average power output in the DF mode. Thus, experimental data confirm that hydrogen produced from a solid propellant grain is as good as commercial hydrogen for use as a fuel in the precombustor of the HF/DF chemical laser.

In the HF laser, hydrogen from the solid gas generator gave comparable experimental results to that of bottled hydrogen. In this mode, the hydrogen, either commercial bottle or solid generator, was the laser cavity fuel. Again, the two sources of hydrogen are equivalent. For all the lasing runs reported in Table 1, the chemical laser device was optimized in the DF mode. Thus, the low power output in the HF mode was attributed to this optimization of the laser in the DF mode.

The apparatus diagramed in Figure 2 and which was used to obtain the experimental data is based on the concept of using an accumulator for the hydrogen gas that is produced from the solid propellant. Also, it should be possible to couple the solid hydrogen gas generator directly to the chemical laser device by designing the solid grain configuration and burning rate to meet the H_2 delivery requirements of the laser system.

Based on the experimental results obtained from the $NaBH_4/Fe_2O_3$ solid hydrogen gas generator, it is apparent that a solid gas generator of this type is feasible and practical as a storable source of hydrogen or deuterium for use in the operation of HF/DF chemical lasers. Compositions that contain $NaBH_4/(NH_4)_2Cr_2O_7$ or $NaBH_4/(NH_4)_2SO_4$ have a much larger theoretical volumetric efficiency and weight percent yield of hydrogen than $NaBH_4/Fe_2O_3$ and, therefore, are potentially attractive as solid hydrogen gas generators. Work is in progress on the preparation and evaluation of solid propellant grains containing these ammonium salts and $NaBH_4$. Only preliminary data have been obtained on the use of $(NH_4)_2Cr_2O_7$ in a solid formulation, but work is continuing on the development of this formulation.

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