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HYDROGEN FLUORIDE CHEMICAL LASER- A DEMONSTRATION OF PURE CHEMICAL PUMPING IN A LAMINAR DIFFUSIVE-MIXING LASER SYSTEM

by

Joseph F. Spinnler

November 1970

U. S. ARMY MISSILE COMMAND
REDSTONE ARSENAL, ALABAMA 35809

Contract DAAH01-70-C-0146, P001

ROHM AND HAAS COMPANY
REDSTONE RESEARCH LABORATORIES
HUNTSVILLE, ALABAMA 35807
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OF PURE CHEMICAL PUMPING III. CHEMICAL PUMPING
IN A LAMINAR DIFFUSIVE-MIXING LASER SYSTEM

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FOREWORD

The work in this report was conducted under Contract DAAH01-70-C-0146, P001 for research on high-energy chemical lasers under the technical cognizance of APL&C, Research and Engineering Directorate, U. S. Army Missile Command, Redstone Arsenal, Alabama. The specific objective of the laser contracts at these Laboratories has been to demonstrate continuous laser action by stimulated emission of hydrogen fluoride pumped solely by the energy released by the homogeneous chemical reaction between hydrogen and fluorine.

Neither the suitability of HF as an emitter nor the efficacy of \( \text{H}_2\text{F}_2 \) pumping had been demonstrated at the time work began. Since then, single-pulse laser action of \( \text{H}_2\text{F}_2 \) has been demonstrated (within the first year's contract), and stimulated emission of HF excited by external means has been reported by other investigators.

Successful chemical pumping by hydrogen and fluorine in a continuous-flow system to excite stimulated emission of carbon dioxide has been reported in the literature. More recently, pumping of \( \text{CO}_2 \) via HF and DF, and the pumping of HF using the energy of chemical reaction alone has been reported, with the latter investigations having been accomplished concurrent with successful results in these Laboratories.

This report is the fourth of a series detailing the laser work in the Redstone Laboratories. The first report (S-139, July 1967) gave the results of gain calculations to determine the energy distribution theoretically achievable in \( \text{HCl} \) and HF as emitters. The second report (S-163, May 1968) described the design and construction of the apparatus, as well as the experimental results obtained during the first year of investigation, culminating in the successful demonstration of pure chemically pumped laser action with hydrogen and fluorine in a single-pulse system. The third report dealt with progress toward a continuous-flow system, including construction of an improved fast-mixing injector to supersede the impinging-jet system used earlier, and various methods of inducing population inversion through introduction of a third species such as NO or fluorine atoms.
This report constitutes the final report on Contract DAAH01-70-C-0146, P001 and, together with the preceding reports under Contracts DAAH-01-67-C-1475 and DAAH01-69-C-0206, covers all the laser work in these Laboratories.

The author wishes to acknowledge the help and advice of our consultants, Professors S. H. Bauer of Cornell University and C. Bradley Moore of the University of California (Berkeley), and the assistance of Professor T. A. Cool of Cornell University for his design of the laminar diffusive burner.

We also acknowledge the assistance of the personnel of the Rohm and Haas Redstone Research Laboratories Engineering Design Group and the excellent job of fabrication of the required equipment by the personnel of the Mechanical Instrument Shop. The assistance of personnel of the Instrument Development Group in detector instrumentation and design and fabrication of the safety system is greatly appreciated. The contributions of technical assistants Messrs. J. W. Clark, W. F. Hooper, and W. M. Davis are also gratefully acknowledged.

Captain William Glass of the Physical Sciences Laboratory, R&E Directorate, U. S. Army Missile Command also assisted in the work covered in this report. Captain Glass's faith in the project, his efforts in obtaining funding, and assistance in the experiments contributed infinitely to the measure of success that was obtained.
ABSTRACT

This report describes results of continuing experiments in a laminar-diffusive mixing laser system. Conclusive evidence for CW coherent laser action of hydrogen fluoride is offered, with pumping energy supplied solely by the energy of chemical reaction. Flow conditions of H₂ and F₂ and other fuels and reactant gases are also presented.
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Section 1. INTRODUCTION

The objective of the laser research at the Redstone Research Laboratories of Rohm and Haas Company was the demonstration of a continuous chemically pumped laser; i.e., the chemical energy of molecular reactions generates population inversion in a reactant species. Most lasers require an external pumping system such as flash lamps, electrical discharge, or arc heating of reactant species. This report describes experiments conducted in these Laboratories using a laminar diffusive-mixing laser system in which continuous-wave (CW) and coherent energy output was observed. Moreover, pumping energy was available only from gaseous reactions \((F_2 + NO, F, F_2 + H_2, H_2)\), a pure chemical laser.

Earlier work in these Laboratories consisted of theoretical analysis of chemical laser systems (1) and design, construction, and experimentation on two types of laser systems - a single-pulse laser system and a turbulent-flow mixing system (2). During the period reported therein, emission characteristic of laser radiation was observed in the single-pulse laser system. Radiation of a similar character was observed in the turbulent-flow system; however, its laser characteristics could not be verified as was done in the case of the single-pulse laser system in ensuing experiments on these systems (3, 4). Rationale leading to design and construction of the laminar diffusive-mixing laser system and initial experimentation are also detailed in Reference 4.

Work reported herein is a continuation of experimentation with the laminar diffusive-mixing laser system. Various flows, mixture ratios of diluents and reactants, and mixing locations in the system under a variety of cavity pressures were investigated. The \(\text{F} \) atom forming reaction

\[
F_2 + NO \rightarrow NOF + F
\]  

(1)

was utilized along with various hydrogenated and deuterated reactant fuels. \(\text{CO}_2\) was also injected without success. Use of \(\text{SF}_6\) was made in order that the system be cryogenically purified.

\(^1\)Numbers in parentheses refer to references at the end of the report.
CW coherent radiation was observed in the case in which pre-cooled fluorine was used. A hydrogen – nitric oxide fuel and sulfur hexafluoride diluent were used in these successful experiments. Low output power prevented characterization of the radiation; however, coherence was verified by determining of the laser cavity.
Section II. EXPERIMENTAL

1. Apparatus

The laminar diffusive-mixing laser system, which includes laser cavity, optical cavity, detector-optical system, gas-metering system, safety system and exhaust system, has been described in detail in References 2 and 3. This system, so described, with minor variations in the plumbing for accommodation of gases and gas mixing locations for various experiments and modifications of cavity optics (variation in mirror reflective surface and focal length), was utilized for the work reported here. The laminar diffusive laser, itself, is shown in Figure 1A. A view of the total system, cavity, optics, reflectors, etc., is shown in Figure 1B.

The optical and detector systems normally utilized are shown in Figure 2. In the experiments in which coherent radiation was detected, the laser cavity mirror (\(M_1\) of Figure 2, the concave mirror) was of 500-mm focal length. This change was made in order that the cavity mode volume might be increased somewhat.

Only the AuGe detector sub-system was useful in the experiments because output intensity was minimal.

A schematic of the flow system is shown in Figure 3. Modifications made involved relocation of various mixing locations for nitric oxide and diluent.

For the experiments in which \(F_2\) and \(NO\) were premixed before admission, this mixing was done in the \(F_2\) manifold of the laser cavity (Figure 4). In the experiments in which the \(F_2\) was cooled, \(F_2\) was allowed to flow first through the rotameter, then through a copper coil immersed in a dry ice trichloroethylene bath, which was adjacent to the laser cavity, and then into the \(F_2\) manifold.

2. Experimental Results

The data from experiments for the period covered by this report have been reduced and the results along with comments and observations tabulated in Table I. Flow rates for the experiments are given in cc/sec at STP for the various gases utilized in the experiments. Where sufficient data were lacking, flows and pressures have been estimated and indicated by ( ). A (+) or (-) indicates that flow was
FIGURE 1.

THE LAMINAR DIFFUSIVE-MIXING LASER SYSTEM

NOT REPRODUCIBLE
FIGURE 2. SCHEMATIC OF THE OPTICAL AND DETECTOR SYSTEMS OF THE LAMINAR DIFFUSIVE-MIXING LASER SYSTEM
F<sub>2</sub> MANIFOLD
MAT'L - 1/2 SCHEDULE MONEL PIPE
.063 DIA DRILL
TWENTY-ONE HOLES
.188 ± .001

MANIFOLD LENGTH = 4 3/4 IN.
WIDTH = 4 1/4 IN.

F<sub>2</sub> MANIFOLD
MAT'L - 0.032 O.D. X 0.044 I.D. MONEL TUBING
NO. REC'D - FIFTY SIX
.0135 DIA DRILL
SIX HOLES, EACH TUBE
.031 TYP

FLUID MIXING TECHNIQUE FOR THE HF CHEMICAL LASER

FIGURE 4. SCHEMATIC OF THE FLUID MIXING TECHNIQUE
FOR THE HF LAMINAR DIFFUSIVE-MIXING LASER
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**BF flow**
- BF flow replaced by N₂ flow.
- N₂ flow on.

**CO flow**
- CO flow on.
- CO flow on, orange radiation around N₂ inlet ports.

** BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.

**NO flow decreased.**
- No radiation at first; then orange radiation appeared.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.

**Radiation decreased in intensity.**
- BF flow off.
- BF flow off, N₂ flow off. No change in cavity observations.

**BF flow increased.**
- BF flow on.
- BF flow increased. Orange radiation around N₂ inlet ports.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.
- BF flow off. Orange radiation increased around N₂ inlet ports.

**NO flow decreased.**
- No change in cavity observations.

**BF flow decreased.**
- BF flow off.
- BF flow off, N₂ flow off. No change in cavity observations.

**BF flow decreased.**
- BF flow off, N₂ flow off. No change in cavity observations.

**BF flow decreased.**
- BF flow off, N₂ flow off. No change in cavity observations.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.

**BF flow increased.**
- BF flow on, orange radiation around N₂ inlet ports.
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**Notes:**
- **SF flow on:** SF flow replaced by Hg flow. Hg MO flow on.
- **Fy flow on:** Hg and MO flow on.
- **Elsenbach signal output observed:** Orange radiation - 9 in MO signal in cavity. MO signal increased. Hg signal decreased. Continuous LASER emission.
- **Increased MO signal:** MO signal in cavity increased. MO signal increased. LASER emission.
- **Reduced MO flow:** MO flow in cavity decreased. MO signal increased. LASER emission.
- **Transit signal observed:** Orange radiation - signal increased. MO signal increased. Continuous LASER emission.

**Initial conditions:**
- SF flow on.
- Hg flow on.
- Hg MO flow on.
- Hg and MO flow on.

**Elsenbach signal output observed:**
- Orange radiation - 9 in MO signal in cavity. MO signal increased. Hg signal decreased. Continuous LASER emission.

**Increased MO signal:**
- MO signal in cavity increased. MO signal increased. Continuous LASER emission.

**Reduced MO flow:**
- MO flow in cavity decreased. MO signal increased. Continuous LASER emission.

**Transit signal observed:**
- Orange radiation - signal increased. MO signal increased. Continuous LASER emission.

**Increased MO signal:**
- MO signal in cavity increased. MO signal increased. Continuous LASER emission.

**Reduced MO flow:**
- MO flow in cavity decreased. MO signal increased. Continuous LASER emission.

**Transit signal observed:**
- Orange radiation - signal increased. MO signal increased. Continuous LASER emission.

**Increased MO signal:**
- MO signal in cavity increased. MO signal increased. Continuous LASER emission.

**Reduced MO flow:**
- MO flow in cavity decreased. MO signal increased. Continuous LASER emission.
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<tr>
<th>T</th>
<th>12.4</th>
<th>12.5</th>
<th>3.1</th>
<th>(35.0)</th>
<th>(35.0)</th>
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Initial conditions - normal feed.

SF, dilute flow on.


SF, flow reduced, N2 flow off. Purple radiation in cavity.

No change in cavity observations.

Reduced SF, flow; SF, flow reduced by N2 flow. Red flow on. Bright purple radiation in exhaust manifold.

Green radiation on CH4 inlet ports; some radiation signal observed on multi-analyzer (Au-Tr-Br.)

Flow of N2 and T4 returned to 60 slm per. Bright blue radiation present in manifold. This transferred to CH4 inlet ports and change in color to orange.

Initial conditions - window purge supply changed from N2/CH4 to CH4 alone. Increased flow rate of CH4.

SF, dilute flow on.

SF, dilute flow replaced by N2 flow. CH4 NO flows on. Fb flow on. SF, flow on in place of N2 flow. Black white radiation in cavity.

SF, flow off. Diffuse blue radiation in cavity.

No radiation observed.


Used N2 to increase cavity pressure (pump inlet bleed). Green radiation appeared at CH4 inlet ports.

Reduced SF, flow (CH4). Blue radiation on exhaust manifold side of cavity.

Reduced SF, flow (Fb). Increased NO flow. Green radiation appeared at CH4 inlet ports. Cut off NO flow and radiation remained at ports.


Reduced SF, flow. Green radiation flashed to CH4 inlet ports.

Used N2 to increase cavity pressure (pump inlet bleed). Green radiation appeared at CH4 inlet ports.

Radiation appeared at exhaust manifold side of cavity.

Reduced SF, dilute flow (CH4). Radiation became green and moved to CH4 inlet ports.

Reduced SF, dilute flow (Fb). Radiation at exhaust manifold side of cavity.

Reduced SF, dilute flow (Fb). Radiation at exhaust manifold side of cavity. Bright green radiation immediately appeared at CH4 inlet ports.

Increased cavity pressure using N2 (pump inlet bleed). Green radiation appeared at CH4 inlet ports.
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NOT REPRODUCIBLE
either increased or decreased but could not be estimated. A tape recorder was used to record pressures, flows, etc., during an experiment, and some flow data were sometimes lost by this process.

The experiments in Table I are listed chronologically. For the purpose of discussion, groups of experiments in which certain common features pertain, will be discussed individually.

a. Initial Experiments

The first experiments after reactivation of the laser system following a period of inactivity between renewal of contracts was for system check-out and for familiarization of Captain William Glass, of the Physical Sciences Laboratory who assisted with later experiments, with equipment, procedures, etc. Conditions approximating those reported in Ref. 3 were utilized for check-out of the system, and are listed in the Table. Results obtained were analogous to those reported previously (Experiments 11-12 January and 27-January 29); i.e., no laser radiation was observed emanating from the cavity; only visible orange radiation was observed, the location of which could be varied by variation in flow.

Conclusions drawn from the previous series of experiments (Ref. 3) were that failure to observe laser radiation might result from the lack of a sufficient number of emitting species in the cavity mode volume.

When attempts were made to increase cavity pressure and, thereby, the concentration of emitters, excessive heating of the manifold resulted in shut-down of the system. This type of experimentation was then deferred until a means of cooling the manifold was devised.

b. Experiments with Premixed F₂–NO

From the previous experiments it was concluded that an insufficient number of emitters were being generated in the laser cavity mode volume. One possible means of increasing this number is the premixing of the F₂ and NO, thereby allowing a greater concentration of F atoms, via Reaction (1) to build up before injection of H₂ into the flow. This approach has also been used by T. A. Cool and R. R. Stephens in this CW laser system (5) and worked satisfactorily in their glass apparatus.
In the experiments run in the laminar diffusive-mixing laser system (Monel® construction), (12-13 Feb)-(15-18 Feb) reaction occurred at the hydrogen inlet pots. It was assumed that the Monel tubes offered a catalytic surface and hence a stabilizing effect on the reaction zone. This reaction zone, as evidenced by orange radiation, could not be moved downstream except under extreme conditions, i.e., no NO flow or high-diluent flow, etc., and with little chance of observing laser radiation existing.

Since it had been concluded previously that the orange radiation (3) was characteristic of deactivated HF, with the result that attainment is highly improbable when it is observed, further experiments in this configuration were discontinued.

c. Experiments with Pre-cooled F₂

Since it had been observed in previous experiments that the orange radiation could be moved downstream, but not readily so in the case of premixed NO + F₂, it was then concluded that it might be best to ensure that the F₂ passed the H₂ inlet ports while in the molecular state and rely on the reaction (1) to generate the first F atoms. Hopefully, with higher concentrations, this reaction plus the two HF reactions

\[ F + H_2 \rightarrow HF + H \] \hspace{1cm} (2)

\[ H + F_2 \rightarrow HF + F \] \hspace{1cm} (3)

could supply sufficient F and activated HF in the cavity-mode volume for laser radiation to be observed. Additionally, catalysis effects should be minimized as the gas flow passes around the H₂ inlet ports.

The flow system was plumbed, as previously described, to accommodate cooling of the F₂ while allowing the diluent to mix with the F₂ in the F₂ manifold of the laser cavity. The NO and H₂ mixing configurations were returned to the normal configuration, i.e., the configuration used before the previous NO-F₂ premix experiments.

CW coherent radiation was first observed under conditions of 15L - 20 Feb. The radiation was observed for periods of 1/2 min. under conditions in which NO flow was rapidly diminishing. The rotameter size used prevented stabilizing conditions satisfactorily.

1 Trademark of The International Nickel Co., Inc., Huntington, W. Va.
Even in later experiments with the smallest diameter rotameter and with smallest flow that could be maintained, the minute amount of NO required still remained a problem.

Attempts were made to maximize the amount of radiation, but insufficient intensity was obtained to allow characterizing of the radiation via the monochromator-detector system. Coherence was verified by detuning the cavity (blocking one of the cavity mirrors).

Under the best operating conditions, 16-23 Feb. 1970, a CW signal could be maintained as long as the $F_2$ and NO flow could be maintained constant (2-5 min.). Estimation of power output was made by comparison of signal of He-Ne laser and approximated 1µ watt for the optical configuration detailed previously with the exception of the 500-mm focal length curved mirror in place of that indicated in the schematic (Fig. 2). The beam splitter was set 5° off the Brewster angle, which for sapphire reflects 0.61% per surface.

Attempts to improve signal output by use of gold surface mirrors 17-4 March were only slightly successful. A greater amount of incoherent radiation was observed along with coherent radiation. Estimated power output was 5µ watts. Again, neither the coherent nor non-coherent radiation was of sufficient intensity to be measured via the monochromator-detector system.

d. **Experiments with Other Fuels**

Since experimentation time was limited and it was agreed that the original objective had been met, i.e., demonstration of CW coherent emission using only commercial bottled gases and with no external energy sources required, a decision was made to investigate other fuels in the laminar diffusive-mixing laser system. These experiments comprised experiments 17-5 March through 19-10 March.

No evidence of coherent radiation was observed using $D_2$, $CH_4$ or $NH_3$. Blue radiation was observed in the case of $D_2$ in contrast to the orange with $H_2$. Blue, green, and violet radiation was observed with $CH_4$ dependent on flow conditions. Deposits of $NH_4F$ and carbon on the cavity windows limited flow conditions that could be attempted with these fuels. Pressure build-up in the fuel manifold system, (the system as designed for $H_2$ fuel requires 0.0135-in. diameter holes in the inlet-port tubes), further restricted the operating range.
e. Concluding Experiments

Since fuels other than \( \text{H}_2 \) appeared less than promising, the remaining experimentation time was utilized in attempts to characterize the observed coherent radiation of HF and to observe 10.6-micron CO\(_2\) radiation via HF and DF energy transfer to CO\(_2\). The former comprised experiments 20-11 March to 22-7 April in which coherent radiation was observed but little increase in power output was realized even though large flow throughputs were used. Apparently the system is limited by pumping capacity and flow capacity when flows of 40 cc/sec STP or greater for \( \text{H}_2 \) and F\(_2\) are attempted. While both reaction products and diluent are cryogenically pumped, this capacity must be exceeded in these flow regimes.

Experiments 23-13 April was an attempt to utilize the energy transfer from excited HF or DF to CO\(_2\). This had been accomplished by Cool and Stephens in their system (5). For experiment 23, the CO\(_2\) was premixed with NO and introduced into the fuel line leading to the laser cavity. The optical system was changed to a 2-mm-hole-coupled hemispherical cavity (gold-surfaced flat and 20.5 in. focal length gold-surfaced concave mirror). KCl windows replaced the sapphire windows of the cavity.

Both \( \text{H}_2 \) and D fuels were utilized; however, no evidence of coherent radiation was observed. Visible orange radiation was observed. This was the concluding experiment; further experimental work on the system, as it has been described, has been terminated.
Section III. DISCUSSION

Upon conclusion of the final experiment in the laminar diffusion-mixing laser system, it appears that realization of high power output from such a system is highly unlikely. The inability to obtain desired concentration levels in the cavity-mode volume is, more than likely, the major deterrent in this system. The device, when coherent radiation was observed, was probably operating near laser threshold. What effects greater pumping rates and fast throughputs might have cannot presently be evaluated.

Doubts currently exist for the suitability of SF₆ as the diluent. It was required in this system because of limited vacuum pumping capacity.

It is significant that coherent CW laser radiation was observed in this system. Output power was miniscule compared with some other types of HF laser systems (6). However, it was in keeping with observations of Cool and Stephens (7), whose system is based on the concept of laminar diffusive mixing even though the axis of observation was different in the two cases; Cool's was axial to the flow while that of this facility's system was transverse to the gas flow.

No further work is contemplated by this facility as all work in this research facility is being terminated.
REFERENCES


HYDROGEN FLUORIDE CHEMICAL LASER - A DEMONSTRATION OF PURE CHEMICAL PUMPING III. CHEMICAL PUMPING IN A LAMINAR DIFFUSIVE-MIXING LASER SYSTEM

Joseph F. Spinnler

November 1970

Technical Report S-272

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Research and Engineering Directorate
U. S. Army Missile Command
Redstone Arsenal, Alabama 35809

(U) This report describes results of continuing experiments in a laminar-diffusive mixing laser system. Conclusive evidence for CW coherent laser action of hydrogen fluoride is offered, with pumping energy supplied solely by the energy of chemical reaction. Flow conditions of H₂ and F₂ and other fuels and reactant gases are also presented.
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