AN EXPERIMENTAL INVESTIGATION OF THE USE OF NITROUS OXIDE IN HYPERSONIC WIND TUNNEL TESTING FACILITIES

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UNITED STATES AIR FORCE
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FOREWORD

This interim technical documentary report was prepared by J. R. Nicholson, E. S. Fishburne, and R. Edse of the Department of Aeronautical and Astronautical Engineering of the Ohio State University on Contract Number AF33(657)-8951, Project 7065, Aerospace Simulation Techniques Research. The research on this task was administered under the direction of the Aerospace Research Laboratories, Office of Aerospace Research, United States Air Force, with Mr. John Goresh, Fluid Dynamics Facilities Laboratory, as Contract Monitor.
ABSTRACT

Various continuous flow hypersonic wind-tunnel testing facilities are discussed in general and the possibility of employing the exothermic decomposition of nitrous oxide to generate high temperature air is considered in detail. The feasibility of establishing high enthalpy air streams by mixing pre-heated nitrous oxide with a hot stream of nitrogen which was heated separately to temperatures ranging from 1033°K to 1264°K was investigated experimentally. The temperatures of the pre-heated nitrous oxide ranged from 875°K to 1055°K. After the decomposition of the nitrous oxide the gas mixture attained temperatures between 859°K and 1807°K. When the decomposition process was incomplete the reacted gas contained a substantial quantity of nitrogen dioxide and nitrogen tetroxide. On the other hand only about 1% of these products was formed when the reaction went to completion. The highest temperature of the reacted products amounted to 1807°K which indicates that nearly 80% of the theoretical temperature rise was obtained.
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SECTION I

INTRODUCTION

The need for laboratory simulation of hypersonic flight and re-entry into the earth's atmosphere has resulted in the continued development of ground-based laboratory facilities. The simulation of the conditions encountered during re-entry and hypersonic flight within the earth's atmosphere requires high-enthalpy air flows. Some of the various methods currently in use to generate high-enthalpy flows with some of their limitations are shown in Table 1.

The experimental facilities fall into two categories: continuous flow facilities and short duration or intermittently operated facilities. Although intermittently operated facilities such as the shock tube, shock tunnel, arc-discharge tube, and the hypervelocity gun range provide many valuable results, for more reliable measurements it is desirable to establish steady flows to permit equilibrium to be established. Stagnation pressures and temperatures must be sufficiently high to permit not only Mach number simulation but also adequate Reynolds number simulation.

Continuous flow facilities include wind tunnels employing electrical resistance heaters, pebble-bed heaters and electrical arc heating. Since no heat transfer is required, combustion gases represent very effective sources of high enthalpies. Frequently their composition differs too much from that of the gas to be studied. However, in the case of nitrous oxide-nitrogen mixtures a composition similar to that of air can be produced. This method has the advantage that pure nitrogen can be heated to higher temperatures than air since there is no oxidizing effect on the heater elements. While air can be heated to about 1500 - 1600°K, pure nitrogen can be heated to temperatures of approximately 3000°K by means of a graphite resistance heater (Ref. 1 & 2).

SECTION II

NITROUS OXIDE WIND TUNNEL

The use of the exothermic decomposition of nitrous oxide as a possible supplementary heat source for hypersonic wind tunnels was first considered by Sabol and Evans (Ref. 3). From a theoretical viewpoint the use of nitrous oxide in hypersonic testing facilities is very appealing. The decomposition is exothermic and releases approximately 20,000 cal/mole (Ref. 4). If one assumes complete decomposition into nitrogen and oxygen a temperature rise of approximately 1500°K can be achieved. The results of thermodynamic calculations for the decomposition of pure nitrous oxide are shown in

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However, the products of the decomposition do not form a gas mixture like air. The mixture is deficient in nitrogen when compared to standard argon-free air (i.e., approximately 79% N₂ and 21% O₂). Addition of high temperature nitrogen to the decomposition products can bring the nitrogen-oxygen ratio to the correct value. To properly evaluate the possibility of employing nitrous oxide it is necessary to determine the rate of the reaction at various pressures and temperatures and the effect of nitrogen addition on the decomposition process.

Previous investigations of the mechanisms of the decomposition of nitrous oxide (Ref. 5-15) have been helpful in designing the apparatus for this study. Since a steady-state wind tunnel configuration must provide a continuous flow of air, information concerning the constant pressure decomposition of nitrous oxide in a flowing system is necessary. In this respect, the preliminary study by Sabol and Evans was inconclusive.

Additional feasibility studies were conducted by Thomas (Ref. 12, 13) who heated a nitrous oxide stream by conventional electrical resistance heaters. It was found that adequate control of the reaction in this heater was extremely difficult if not virtually impossible. The nitrous oxide decomposed in the eddy region behind the heater wires and transferred energy to the wires causing them to melt. Subsequent experiments were conducted with the electrical resistance heater employed primarily as a nitrous oxide pre-heater in conjunction with an alumina pebble-bed as a reaction chamber. Adequate control of the reaction could be maintained with this configuration. Indirect measurements indicated local temperatures in excess of 2000°K. However, the formation of large quantities of intermediate oxides of nitrogen, such as NO and N₂O, created an objection to the use of nitrous oxide in hypersonic wind tunnels. No specific gas analyses were made.

The results of subsequent experiments conducted at this laboratory (Ref. 8, 14) have indicated that at a pressure of 11.2 atmospheres approximately 5% by volume of NO and N₂O is formed. This percentage may not be a severe limitation since equilibrium air at 2500°K has approximately 2 to 3% NO (Ref. 16). Experiments also were conducted with a pre-mixed system of N₂O + .88 N₂. Again 3 to 5% N₂O and NO appeared in the products. During all of the experiments a phenomena was observed which was very similar to flash back which occurs when the gas flow of burner flames is reduced. Theoretical considerations indicate that the rapid decomposition resembles a thermal explosion. However, the decomposition did not travel upstream. In view of this behavior it is preferable to preheat flowing nitrous oxide to a temperature just below the explosion limit (900-1000°K) and then mix it with nitrogen which has been heated to 3000°K. The temperature of the products of the reaction is approximately 2500°K. Of course the reaction must be complete before the gas mixture reaches the throat of the nozzle. Nitrogen temperatures of around 3000°K have been obtained on a continuous basis with a spiral graphite heater (Ref. 1-2). If the preheated nitrous oxide can be introduced close to the nozzle inlet the heat loss to the container walls may be reduced.

Calculations of the temperature that can be obtained by the procedure were made for various nitrogen and nitrous oxide initial temperatures. The results are shown in Figure 2. Since the amount of nitric oxide formed during the decomposition does not appear to differ much from that of equilibrium
air, it was assumed for the purposes of the calculations that the resulting mixture had the composition of equilibrium air. The thermodynamic data for the calculations were obtained from (Ref. 4 and 17).

Since nitrous oxide cannot be heated to temperatures higher than 1000°K without rapid decomposition, the final gas temperatures that can be established depend primarily on the temperatures to which the nitrogen can be heated. The final gas temperature can be maintained at a fairly constant value in a continuous-flow facility employing nitrous oxide. Furthermore, the gas flow is basically contaminant free. In contrast, gases heated in a pebble-bed contain a significant amount of contamination and the stagnation temperature decreases steadily.

SECTION III

EXPERIMENTAL

To avoid decomposition of nitrous oxide in the heater, separate heaters were used to heat flowing nitrogen and flowing nitrous oxide. The decomposition of the nitrous oxide occurs during the mixing process. Previous investigations at this laboratory (Ref. 8, 14) gave evidence of a substantial effect of the tube walls on the reaction. It was not ascertained whether the decomposition at the wall was catalytic in nature or simply resulted from the high wall temperature. Since the flow was introduced in a tangential manner, it is possible that because of the helical nature of the flow a considerably higher gas temperature existed in the region adjacent to the walls. In all cases much of the energy of the reaction was transferred directly to the walls so that all of the observed temperature rises were much less than the theoretically calculated values.

As a result of these experiments it was felt that it would be advantageous to produce a decomposition reaction in a high temperature nitrogen stream. Provisions were made in the design to permit investigations at pressures up to 50 atmospheres. An existing high pressure Bunsen burner was modified for use in this study.

Various methods of heating the nitrogen and nitrous oxide streams were considered. First, a coaxial heater was tried. A single heater element was wrapped around the central tube so that both streams were heated simultaneously. However, this system did not allow adequate control over each individual stream. Therefore, two separate heaters were constructed.

The nitrous oxide heater consisted of a 36 inch high-purity alumina tube with a one inch ID. The tube was wrapped externally with 12 gauge resistance wire. The resistance element was embedded in alumina cement to provide good thermal conductivity as well as to provide structural support for the element. The entire heater assembly was placed within a water cooled stainless steel jacket and thermally insulated from the jacket with loose fibrefrax. It was found that rather poor heat transfer rates were obtained with this system. Therefore the heater was converted to a pebble-bed type.
heater by placing small alumina pebbles in the heater section. This arrange-
ment increased the heat transfer rates considerably. The temperatures of
nitrous oxide at the exit of the heater was measured with a steel sheath
platinum-13% rhodium thermocouple.

Considerable difficulty was encountered in obtaining a heater for the
nitrogen system. Initially, a heater similar to that for nitrous oxide was
employed. However, since this heater was installed horizontally, whereas
the nitrous oxide heater was installed vertically, pebbles could not be
used. The heater wires burned out several times because the heat was not
conducted away from the wires at sufficient rates. In an attempt to reduce
the temperature gradient between the wires and the gas stream the heater
coil was placed inside the alumina tube. Unfortunately, it was impossible
to protect the exposed wire from mechanical and chemical damage.

Temperature measurements were made at the exit of the heater and at
the entrance to the mixing chamber. It was found that a significant amount
of heat was lost in this region. For example, with an outlet temperature
at the heater of 1350°K a temperature of approximately 1050°K was obtained
at the outlet of the transfer tube. Since a gas temperature of 1350°K
apparently corresponded very closely to a wire temperature near the failure
point, a significant increase in the maximum nitrogen temperature above
1050°K was impossible. In an attempt to reduce the heat loss to these sur-
roundings, a heater element was added to the transfer tube. This modifica-
tion increased the maximum nitrogen temperature by approximately 100°K.
Since nitrogen temperatures of at least 1300°K were needed this mode of
heating was abandoned.

It was decided to try a carbon element heater since carbon can be heated
to 3000°K in oxygen-free gases. The usefulness of a carbon resistance ele-
ment has been demonstrated (Ref. 1). Because of low resistance of the car-
bon element, a high current power supply had to be used to generate suffi-
cient power in the heater element. Shreeve, et. al., (Ref. 1) concluded
that a heater with a spiral flow passage machined into a graphite rod which
was slipped into a thin graphite cylinder would produce maximum heat trans-
fer. To avoid the complex construction of such heater elements it was de-
cided to try a straight carbon tube. Carbon was chosen instead of graphite
because of its higher specific resistivity (nearly four times as high as
that of graphite). Consequently, for a given geometrical configuration and
current level the carbon element can handle four times as much power. A
sketch of the element and the electrode clamp is shown in Figure 3.

The outside diameter of the carbon element is 1/2 inch. A 1/4 inch
diameter hole was drilled axially through the rod. The effective resistance
of the rod and clamp assembly was approximately 0.05 ohm. The total cur-
cent loading on the elements varied from about 2100 amhs/in.2 for an 18-inch
heater to 2300 amhs/in.2 for a 14-inch heater. These current densities
are somewhat above the recommended loading current but in order to obtain
sufficient power input to the gas these current densities were necessary.
Since the total length of the tube varied from 14 to 18 inches while the
heated length ranged only from 11 to 15 inches, calculations of the Reynolds
number of the gas flow indicated that nitrogen flow rates up to approximately
200 cc/sec would be laminar. Flow rates of 157 cc/sec produced appreciably
lower temperatures at the heater exit than did higher flow rates; however, when the flow rate was increased the maximum gas temperature increased only about 100 K. When the flow rate was increased beyond about 600 cc/sec the exit temperature decreased.

Details of the nitrogen heater and nitrous oxide heater are shown in Figure 4. The two gas streams are brought together perpendicularly to each other in a conical nozzle. The gas mixture then passes into a stainless steel baffle system to insure proper mixing of the two streams. The reactor tube consists of a 1-inch ID alumina tube five inches long. The reactor tube is wrapped with heater wire to reduce the heat losses from the reactor to the ambient surrounding. A platinum/platinum - 13% rhodium thermocouple was positioned at the outlet of the reactor to measure the reactive gas temperature. In addition, a vacuum sample system was provided to withdraw gas samples from the stream at the axial position of the thermocouple. Subsequent experiments were conducted with a thermocouple probe and a gas sample probe which could be positioned at various axial positions above the outlet of the baffles.

SECTION IV

EXPERIMENTAL RESULTS

Experiments were initiated at atmospheric pressure with a total volume flow rate of one liter/sec. The individual flow rates of nitrous oxide and nitrogen amounted to 532 cc/sec and 468 cc/sec respectively. Temperature measurements and gas samples were taken at the exit of the zirconia mixer. Within the limited range of nitrogen temperatures possible with the resistance heater there was no appreciable decomposition at this probe position and flow rate. Since the distance from the point of mixing of the nitrogen and the nitrous oxide stream to the probe was very small (approximately 1/4 inch) the reaction times were quite small. In addition, the surrounding air provides an effective reaction quenching mechanism; hence, no apparent decomposition was observed. In order to produce decomposition a one-inch ID alumina tube 5-1/4 inches long was placed at the outlet of the zirconia mixer. This tube effectively prevented entrainment of air by the nitrous oxide-nitrogen mixture. The reactor tube was wrapped with an electrical resistance heater element and insulated to reduce the heat loss from the gas.

With this system it was observed that a homogeneous flow was extremely difficult to obtain. Samples taken in a given axial position at various radial positions from the tube centerline showed that mixing of the two gas flows was incomplete. To insure complete mixing of the nitrous oxide and nitrogen streams a steel baffle system was placed within the alumina reactor tube at the outlet of the zirconia mixer. This baffle consisted essentially of three plates positioned normal to the flow such that the increased turbulence would enhance the mixing of the gases.
Another factor to be considered is the residence time of the gas in the reactor. For a total flow rate of one liter/sec and at approximately 1100°K the residence time of the gas was much less than the calculated decomposition half-life of nitrous oxide. For optimum results, considering the nitrous oxide decomposition half-life and the geometrical configuration of the reactor, a nitrous oxide flow rate of 80 cc/sec was found to provide acceptable residence times. However, this value corresponds to a nitrogen flow rate of 70 cc/sec which was considerably less than was desirable because of the heat transfer rates of the nitrogen heater system. It was found that a nitrogen flow rate of 141 cc/sec provided a good solution to the problem with regard to maximum residence time and optimum heat transfer characteristics for the nitrogen heater element.

It was impossible to obtain an appreciable amount of decomposition of the nitrous oxide without the reactor tube heater section. Because of the secondary heating of the mixture it was impossible to determine the precise state at the entrance of the reactor tube. The mixer and associated heater played a very important role in the onset of significant decomposition. In some instances (e.g., when failure of the nitrogen heater caused a reduction in the nitrogen temperature) the decomposition could be maintained on the steady state basis. During these experiments the nitrous oxide temperature before mixing remained essentially constant, indicating that no reactions were occurring in the nitrous oxide heater section.

The critical pre-heat temperature for the nitrous oxide at one atmosphere was found to be in the temperature range between 973°K and 1023°K which is essentially in agreement with the results obtained in previous investigations of the constant pressure decomposition of nitrous oxide at 11.2 atmospheres (Ref. 14). At temperatures above 1023°K with constant power input to the nitrous oxide heater a steadily increasing exit temperature was observed which was indicative of a reaction occurring in the pebble-bed nitrous oxide heater. These temperature rises were observed very carefully and at no time caused a failure of the nitrous oxide heater.

By means of the heater reactor tube it was possible to obtain an apparent decomposition "flame". The affluent gases were luminous, a characteristic which has been observed previously (Ref. 8 & 14). However, no distinct cone could be seen. Typical photographs of the decomposition flame are shown in Figures 5 and 6. These figures also show the effect of increasing nitrogen concentration at a nearly constant volume flow rate. For a mixture containing approximately 90% nitrous oxide the liminosity of the flame is quite intense. The intensity decreases as the nitrogen flow is increased. The color of the liminous reaction zone changed also. With nearly pure nitrous oxide the reaction zone looks deep reddish-orange, while with increasing concentrations of nitrogen it becomes very pale and yellow-green. The decompositions at lower initial temperatures were accompanied by a pale yellow-green reaction zone while the higher temperature reactions were characterized by a reddish-orange radiation.

Many of the experiments were conducted with the thermocouple and the gas sampling probe positioned at the outlet of the reactor tube. The results of these experiments are shown in Table 2. In these experiments, the
maximum temperature was 1317°K and the decomposition was approximately 80% complete. The measured temperature rise was less than 400°K whereas the theoretical temperature rise should have been 1000°K. Obviously, much heat is lost through the reactor tube. Subsequent experiments in which the thermocouple and gas sample probe were positioned in an axial position just downstream of the baffle system indicated much higher temperature rises as well as more nearly complete decompositions. An important factor in these reactions was the relatively high power input to the reactor tube producing higher baffle temperatures. This high power input produces a higher temperature rise as well as an increased reaction rate. This technique led to a maximum measured temperature of 1807°K at a position approximately 1.0 inch upstream from the reactor exit.

The lowest concentration of N₂O remaining in the reacted gas was observed for a final temperature of 1651°K with 2.34% N₂O by volume. The samples of gas taken at the outlet of the baffle system 1/2 inch, 1 inch, and 1-1/2 inch downstream, respectively, gave interesting results. As we proceed downstream the temperature increases while the nitrous oxide concentration decreases. In the initial stages of the decomposition reaction large amounts of NO or NO₂ are formed which decrease as the reaction goes more nearly to completion. At a temperature of 1224°K a maximum equilibrium temperature concentration of NO₂ + N₂O₄ was noted. At a temperature of 1611°K the lowest NO₂ concentration was observed and the reaction had proceeded approximately 96% to completion. This effect had been observed previously by Kaufman et. al. (Ref. 18).

SECTION V

CONCLUSIONS

The feasibility of employing nitrous oxide as an energy source for a hypersonic wind tunnel has been demonstrated by mixing a nitrous oxide stream of 950°K with a nitrogen stream heated to approximately 1200°K. The temperature of the mixture then increased by approximately 1000°K because of the heat release by the nitrous oxide. A major drawback to the use of nitrous oxide lies in the formation of the intermediate oxides of nitrogen. In the present arrangement the formation of these nitrogen oxides is quite small. Since the reaction occurred nearly to completion at the nitrogen temperature levels involved (1100 - 1200°K), and the residence times considered (approx. 0.02 second), the design of a system which utilizes the spiral graphite heater at nitrogen temperatures to 2000°K would result in extremely short reaction times. The reaction chamber could be small thereby reducing the heat losses from this chamber. On the other hand, the steady-state use of nitrous oxide in a wind tunnel system is very costly. To obtain high stagnation pressures (above 1000 PSI) nitrous oxide at high pressures must be available.

In contrast, a system utilizing the separate heating of oxygen and nitrogen would be cheaper. It appears to be entirely possible to heat pure
oxygen to temperatures of 1500°K on a steady state basis with chrome-nickel-aluminum alloy resistance wires, while nitrogen can be heated easily to 3000°K with spiral graphite heaters. Upon mixing, air temperatures of approximately 2600°K can be produced on a steady-state basis with economical operation. This temperature is higher than the highest reached thus far with the nitrous oxide - nitrogen system. Various mixture temperatures for preheated oxygen-preheated nitrogen systems are given in Figure 7. Although the high cost of commercially available nitrous oxide is somewhat offset by the lower electrical energy requirements for the nitrous oxide-nitrogen system, it seems that the operational ease and the simpler containment problem in the separate heating of nitrogen and oxygen would more than compensate for this advantage.
REFERENCES


4. JANAF Interim Thermal Chemical Tables, Thermal Laboratory, The Dow Chemical Co., December, 1960.


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**TABLE 1**

COMPARISON OF SOME HYPERSONIC TESTING FACILITIES
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$^a$ Probe positioned at exit of reactor tube

$^b$ Probe position 1 inch from exit of reactor tube

$^c$ Maximum observed temperature = 180°C = Probe position approximately 1.0" from exit

**TABLE 2**

RESULTS OF EXPERIMENTS WITH PRE-HEATED NITROUS OXIDE AND PRE-HEATED NITROGEN
**FIGURE 1. INITIAL vs FINAL TEMPERATURE OF A CONSTANT PRESSURE $N_2O$ DECOMPOSITION**

$N_2O \rightarrow N_2 + aO_2 + bO$
Figure 2. Mixture temperature versus initial nitrogen temperature (nitrous oxide and 0.88 nitrogen).
FIGURE 3. CARBON HEATER ELEMENT
FIGURE 4. CROSS-SECTIONAL VIEW OF EXPERIMENTAL APPARATUS
FIGURE 6. NITROUS OXIDE - NITROGEN DECOMPOSITION FLAME
TOTAL FLOW RATE ~ 300 CC/SEC

20% NITROUS OXIDE

50% NITROUS OXIDE
FIGURE 7. MIXTURE TEMPERATURE VERSUS INITIAL NITROGEN TEMPERATURE
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AN EXPERIMENTAL INVESTIGATION OF THE USE OF NITROUS OXIDE IN HYPERSONIC WIND TUNNEL TESTING FACILITIES

Scientific Interim

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Technical Report # 5

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Various continuous flow hypersonic wind-tunnel testing facilities are discussed in general and the possibility of employing the exothermic decomposition of nitrous oxide to generate high temperature air is considered in detail. The feasibility of establishing high enthalpy air streams by mixing pre-heated nitrous oxide with a hot stream of nitrogen which was heated separately to temperatures ranging from 1033°K to 1264°K was investigated experimentally. The temperatures of the pre-heated nitrous oxide ranged from 875°K to 1055°K. After the decomposition of the nitrous oxide the gas mixture attained temperatures between 859°K and 1807°K. When the decomposition process was incomplete the reacted gas contained a substantial quantity of nitrogen dioxide and nitrogen tetroxide. On the other hand, only about 1% of these products was formed when the reaction went to completion. The highest temperature of the reacted products amounted to 1807°K which indicates that nearly 86% of the theoretical temperature rise was obtained.
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### KEY WORDS

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<tr>
<th>Nitrous oxide</th>
<th>High enthalpy wind tunnels</th>
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