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Handling Considerations of Nitrous Oxide in Hybrid Rocket Motor Testing

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Nitrous Oxide has many beneficial properties that make it a good choice for use in hybrid rocket motors. The large quantities and potentially high pressures used in rocket motors present unique hazards that are not generally found in the standard industrial and medical uses of nitrous oxide. Through years of hands on use, and research into the properties of nitrous oxide, SpaceDev has created a set of guidelines on how to design, clean, and inspect systems using nitrous oxide. This paper presents the nitrous oxide handling precautions and procedures employed by SpaceDev in the testing of hybrid rocket motors.

Nomenclature

AMROC	=	American Rocket Corporation
BAA	=	Broad Agency Announcement
HTPB	=	Hydroxyl-Terminated Polybutadiene
LOX	=	Liquid Oxygen
N2O	=	Nitrous Oxide
PMMA	=	Polymethyl Methacrylate

I. Introduction

NITROUS Oxide has recently seen a lot of interest as an oxidizer for hybrid rockets. Nitrous oxide has many properties which make it a good choice for a variety of different mission applications. It is non-toxic, long term storable, has good density, a high vapor pressure, and has good overall performance. However, nitrous oxide is exothermic, and is an oxidizer. As with all oxidizers and exothermic materials, proper care needs to be taken when handling nitrous oxide.

Engineers using nitrous oxide for rocket propulsion systems need to recognize that it does have the potential to rapidly decompose when exposed to ignition sources or, if contaminated with fuels, burn. Both the rapid decomposition and / or burning can lead to explosions from pressure vessel failures. A decomposition reaction of nitrous oxide can increase the pressure in a tank or line by over an order of magnitude in a fraction of a second. However, it is possible to avoid nitrous oxide decomposition through the use of proper storage and handling techniques. It is essential to isolate nitrous oxide systems from possible ignition sources and to keep contaminates out of the system. When proper procedures are followed nitrous oxide is one of the safer oxidizers used today in rocket motors.

This paper highlights the training, handling and safety procedures employed by SpaceDev in the testing and operation of hybrid rocket motors. This paper is intended only to highlight how SpaceDev handles nitrous oxide and is not intended to be used as a comprehensive guide on the design of nitrous oxide systems or nitrous oxide safety. While SpaceDev has done its best to verify that all the information contained in this paper is factually correct, SpaceDev assumes no liability for errors contained within this paper.

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II. SpaceDev's Nitrous Oxide Hybrid Motor Testing Heritage

A. AMROC

SpaceDev's background and expertise in hybrid propulsion technology was originally derived from the knowledge base produced by the American Rocket Company (AMROC). In 1998 SpaceDev obtained the technical rights, proprietary data and patents produced by millions of dollars worth of hybrid rocket motor research conducted by AMROC. This included all procedures and over a decade of information on the use of liquid oxygen and nitrous oxide in hybrid motor testing.

AMROC designed and hot test fired a wide variety of hybrid rocket motors, utilizing non-toxic propellants, including nitrous oxide. AMROC completed approximately 300 hybrid motor tests from 100 to 250,000 pounds of thrust. Figure II-1 depicts the 250,000 pound thrust motor which was the largest hybrid motor hot-fired by AMROC.

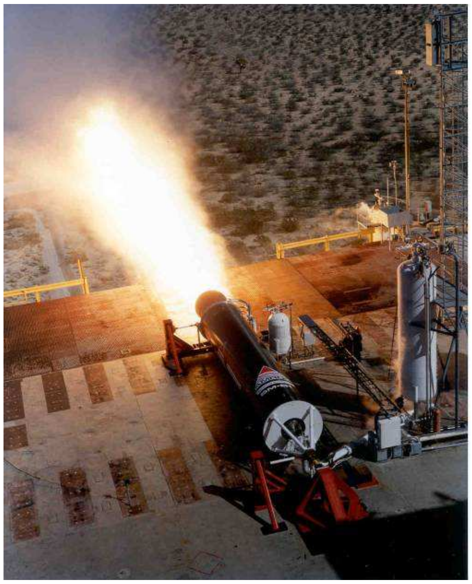

Figure II-1: The 250,000 lb thrust motor, utilizing LOX and HTPB

B. Hybrid Maneuvering and orbital Transfer Vehicle

SpaceDev has continued AMROC's hybrid work and has advanced the use of nitrous oxide in hybrid rocket motors. In 2000 SpaceDev's subsidiary Integrated Space Systems was issued an economic development grant from the State of California to begin development of a small satellite orbital transfer vehicle based on AMROC's nitrous oxide hybrid rocket technology. The grant allowed SpaceDev to construct and successfully fire a Polymethyl Methacrylate (PMMA) / nitrous oxide motor.

The work started under the grant from California was continued under a BAA award from the National Reconnaissance Office, Office of Space Launch Advanced Programs Division. Under the BAA Program SpaceDev conducted a total of 13 different hot fire tests on 8 different motors.

Upon completion of the NRO BAA SpaceDev secured a Phase I and Phase II SBIR from AFRL to continue development of the MoTV nitrous oxide hybrid propulsion technology. Under the AFRL phase II SBIR SpaceDev conducted a total of 14 hot fire tests on four different motors. The four motors had a combined firing duration of over 300 seconds. The prototype and a test firing are shown in Figure II-2.

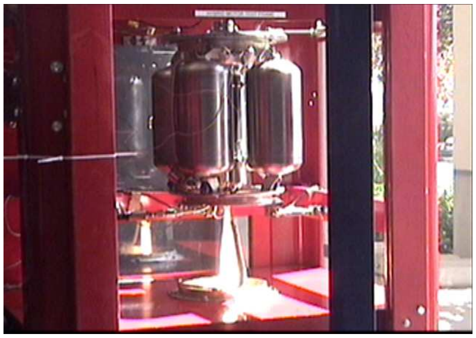

Figure II-2: MoTV-1 Prototype MoTV using N2O / PMMA

C. SpaceShipOne

The SpaceShipOne motor, developed by SpaceDev and Scaled Composites, used much of same the technology developed through AMROC and the MoTV program to build a human rated 15,000 pound thrust motor using nitrous oxide and HTPB. Nitrous oxide was chosen for SpaceShipOne's hybrid motor because of its safety advantages over liquid oxygen, nitrogen tetroxide, and hydrogen peroxide. SpaceShipOne is the largest nitrous oxide hybrid motor ever flown and is the only hybrid rocket to ever propel a human to the edge of space.

Figure II-3: SpaceShipOne Test Flight

D. SpaceDev SLV Program

Based upon the experience gained in the SpaceShipOne program, SpaceDev was able to secure a SBIR from the Air Force Research Laboratory (AFRL) for development of a nitrous oxide / HTPB hybrid to support an all hybrid Small Lunch Vehicle (SLV). Currently the program is in a Phase III and development of the upper stage motor is well underway. Six separate motor tests have been completed and more testing is set for the near future. Figure II-4 shows the testing of the Hybrid Upper Stage (HUS) motor at SpaceDev's hybrid rocket test facility in San Clemente CA.

Figure II-4 Test Firing of Streaker SLV Hybrid Upper Stage Using Nitrous Oxide and HTPB

III. The Handling and Properties of Nitrous Oxide

A. Health Effects from Inhalation of Nitrous Oxide

Nitrous Oxide is non-toxic and widely used by dentists to calm patients undergoing various procedures. Nitrous oxide is commonly administered in dentists' offices at up to a 50% oxygen to 50% nitrous oxide ratio[1]. Primarily the short term dangers involved with gaseous nitrous oxide center around possible suffocation in large concentrations and impaired judgment caused by the euphoric feeling of nitrous oxide. As everyone is different, the amount of nitrous oxide in air required for feelings of euphoria will vary from person to person. If using nitrous oxide in a confined space SpaceDev requires the use of an oxygen sensor to prevent personnel from inhaling dangerous concentrations of nitrous oxide. As with any industrial chemical, or process, SpaceDev requires two people to be present when handling nitrous oxide. Other short term exposure effects and OSHA Long term exposure guidelines are published in the nitrous oxide material safety data sheets provided by the manufacturers[2,3].

B. Reactivity

Nitrous Oxide is both exothermic and an oxidizer, however it is generally stable in both liquid and gaseous forms at lower temperatures and pressures. Nitrous oxide will burn with fuels at almost any temperature and pressure as long as it has an ignition source. As with all oxidizers, Nitrous oxide lines and tanks must be cleaned to insure that no fuels or catalysts are present. The presence of even a small amount of a fuel or catalyst material in tanks and lines can greatly reduce the energy threshold required for initiation of the decomposition reaction. Nitrous oxide can form explosive mixtures with many common hydrocarbons used as oils and lubricants as well as other fuels. SpaceDev's procedures highlight the importance of keeping nitrous oxide lines clean from contaminates, and away from possible ignition sources.

At temperatures exceeding 1200°F (650°C) nitrous oxide can begin to rapidly exothermically decompose at pressures as low as 1 atmosphere without any contamination. At pressures above 200psi, and ambient temperatures, it is possible to start a self sustaining reaction in a tank or large diameter pipe with an ignition source[4]. While nitrous oxide is capable of starting the decomposition reaction on its own, generally an ignition source such as a spark, flame, compressive heating or other heat source is required to start the reaction. Nitrous Oxide is safest when stored in its pure liquid form at low temperatures and pressures. SpaceDev stores nitrous oxide at approximately 0 degrees F to minimize the risks of a decomposition reaction.

C. Common Ignition Sources

A nitrous oxide reaction can be started from virtually any heat source with sufficient energy. Standard ignition sources such as open flames, static discharge, sparks, compression heating or even an overheating pump need to be kept away from nitrous oxide.

IV. Design of Nitrous Oxide Systems for Hybrid Rocket Systems

A. Material Compatibility

SpaceDev's preferred material for tanks, tubing, piping etc in nitrous oxide service is 304 or 316 stainless steel. Materials such as copper, nickel, platinum, and other common catalysts are avoided by SpaceDev because of possible catalytic effects at higher temperatures and pressures. All tanks and plumbing used by SpaceDev conform to standard ASME pressure vessel and piping codes or equivalent standards for ground test equipment.

Materials that are not compatible with oxygen should never be used with nitrous oxide in rocket propulsion systems. Many materials that are compatible with liquid oxygen can be used in nitrous oxide service. However, SpaceDev checks all materials, such as o-rings, valve seats, etc for nitrous oxide compatibility prior to installation in nitrous oxide service. Just because a material is compatible with liquid oxygen does not mean that it is automatically compatible with nitrous oxide. If no data is available on a particular material, SpaceDev follows standard industry practices for evaluating material compatibility.

B. Electrical Conductivity of Nitrous Oxide

The electrical conductivity of nitrous oxide is low enough that with flowing nitrous oxide it is theoretically possible to produce a large enough static discharge to initiate a decomposition reaction. SpaceDev's engineers take nitrous oxide's relatively low electrical conductivity into account when designing nitrous oxide systems. The easiest way to avoid static discharge is to construct nitrous oxide tanks and lines out of conductive materials which are properly grounded. For composite pressure vessels, SpaceDev engineers employ various methods to dissipate the possible static charge.

C. Rust Contamination Issues

Most pressure vessels used in the production and transport of nitrous oxide by gas suppliers are carbon steel. To prevent rust contamination issues in sensitive components, SpaceDev uses stainless steel for all rocket propulsion test applications involving nitrous oxide. To help prevent contamination SpaceDev places stainless steel filters on the inlet to all stainless steel plumbing. Because of the significant use of carbon steel tanks in nitrous oxide production it is difficult to remove all traces of rust from nitrous oxide systems. Therefore SpaceDev does not allow systems in service for nitrous oxide service to be used with hydrogen peroxide or other chemicals which may react with rust contamination without proper cleaning and inspection.

D. Pumping of Nitrous Oxide

Nitrous Oxide is pumped every day throughout the world. However, some of the most significant accidents involving nitrous oxide have been a result of a pump malfunction coupled with a breakdown in properly following safety procedures. Because nitrous oxide is shipped and stored at its saturation point, it is fairly easy to cavitate a pump. If a pump does run dry and overheats, the pump itself can form an ignition source leading to a catastrophic explosion. One such explosion occurred in 2001 at a Linde filling station[5].

Base of Trailer and Associated piping *(Image Source pg 12 ref 5)* | Cab of Truck *(Image Source pg 15 ref 5)* | Trailer's Tank *(Image Source pg 16 ref 5)*

Figure IV-1: The Remains of a N2O Delivery Truck after an Explosion Caused by a Pump Malfunction

When a system involves the pumping of nitrous oxide, SpaceDev uses redundant safety measures to verify that the pumps are pumping liquid properly. Additionally, SpaceDev's pumps are pre-chilled prior to turning on the pump to help avoid boiling / flashing of nitrous oxide in the pump.

E. Pressurization of Nitrous Oxide Tanks

The pressurization of nitrous oxide presents hazards unique to nitrous oxide. Nitrous oxide's high vapor pressure combined with its exothermic decomposition reaction must be taken into consideration when pressurizing tanks of nitrous oxide. Accidents have occurred by rapidly pressurizing nitrous oxide at elevated temperatures and pressures[6,7].

The rate of pressurization should be maintained at relatively low levels to avoid an exothermic decomposition reaction started through compression heating. SpaceDev normally limits the pressurization of nitrous oxide to a maximum ramp rate of 20 psi per second. However, this limit may be reduced even further in certain cases. For pressurized nitrous oxide systems, thermal calculations can be preformed to determine how fast the temperature will rise in the ullage prior to setting pressurization rates. For more information of the ramp rate see reference 4.

F. Warming of Nitrous oxide

For some rocket applications it may be desirable to heat nitrous oxide from storage conditions of approximately 0° F to ambient temperature or above. Nitrous oxide's liquid density is highly dependent on temperature. It is critical that when warming up nitrous oxide there be sufficient ullage for the liquid nitrous oxide to expand in to. SpaceDev requires that all tanks and lines with potentially trapped volumes to be equipped with pressure relieving devices such as relief valves and/or burst discs. Failure to plan for the expansion of the liquid volume can lead to a rapid pressure rise capable of failing pressure vessels. As Figure IV-2 shows, liquid nitrous oxide can expand approximately 20% by volume between bulk delivery temperature and room temperatures.

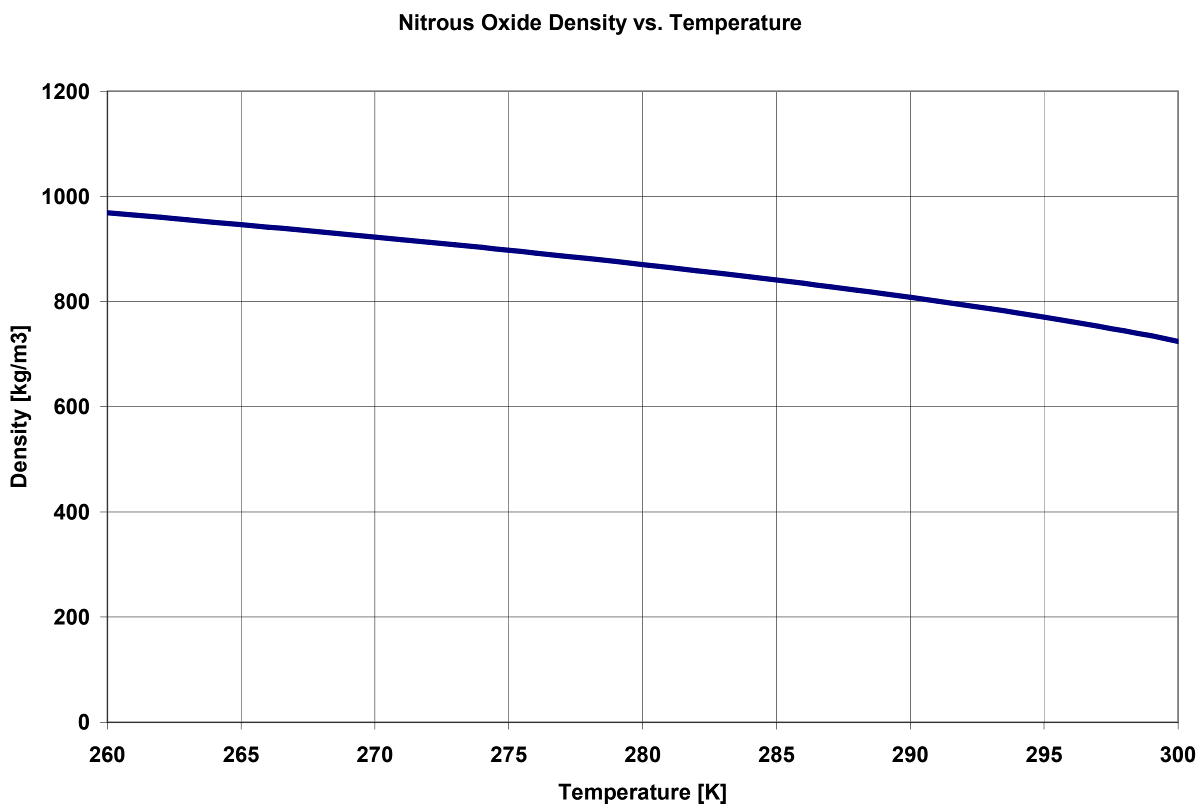

Figure IV-2: Density of Nitrous Oxide as a Function of Temperature at Saturation Conditions[8,9]

SpaceDev's preferred method for warming nitrous oxide is to fill a run tank with sufficient ullage volume and allow it to slowly warm up to the desired temperature or through the use of an ambient air heat exchanger. If an active heating system must be used, such as for testing in colder climates during the winter, SpaceDev runs the nitrous oxide through a heat exchanger with a uniform wall temperature such as a coil submerged in a water bath. SpaceDev never exposes nitrous oxide lines, tanks, ect to open flames or other concentrated heat sources. Hot spots in heat exchangers can lead to catastrophic failures even with liquid flow. SpaceDev has conducted several tests on nitrous oxide heat exchangers in association with Purdue University and has successfully demonstrated that it is possible to start a decomposition reaction with liquid nitrous oxide in tubing under a ¼" diameter by exposing it to an extreme, highly concentrated heat source as shown in Figure IV-3.

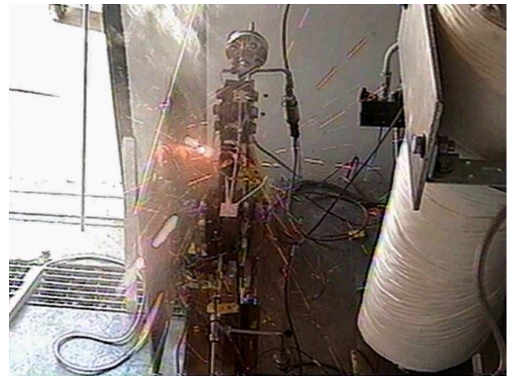

Figure IV-3: SpaceDev Nitrous Oxide Heat Exchanger Experiment Conducted At Purdue University.

V. Preparation of Components for Nitrous Oxide Service

A. Cleaning and Handling of Parts

SpaceDev follows strict cleaning procedures and material compatibility checks for nitrous oxide systems. SpaceDev uses procedures based off of cleaning standards developed for the cleaning of liquid oxygen systems. SpaceDev's procedure for the cleaning of parts going into use in nitrous oxide systems is based off ASTM G93[10], CGA G-4.1[11], and MIL-STD-1330D[12].

All SpaceDev parts go through a four step cleaning process:

- Pre-cleaning
- Deep cleaning
- De-ionized water rinse
- Dry with nitrogen

SpaceDev's pre-cleaning consists of removing all dust, or other lose contaminates with a brush or other suitable method. Once, loose particles are removed an oxygen cleaning agent per either MIL-STD-1330D or ASTM G-127[13]is used to thoroughly clean the part by hand. The part is then rinsed with clean water.

SpaceDev's preferred method for deep cleaning parts is through the use of an ultrasonic cleaner per relevant standards such as ASTM G-131[14] or MIL-STD-1330D. Parts too large to be cleaned in an ultrasonic cleaner, are cleaned by following the processes outlined in ASTM G93 for the specific type of part.

SpaceDev uses de-ionized water[15] to rinse the cleaned parts, each part is rinsed and soaked in de-ionized water 3 times to ensure that the oxygen cleaning agent is fully removed from the part. The part is then dried with clean nitrogen gas.

B. Inspection of Cleaned Parts

SpaceDev uses a three part inspection process on all components entering nitrous oxide service per ASTM G93.

- Visual
- Wipe
- UV Lamp

SpaceDev's initial inspection consists of a visual inspection looking for obvious loose contaminates, grease, etc. If the part passes the initial visual inspection, sample areas are wiped with a white lint free cloth. The cloth must come out clean to pass and move on to UV inspection. Finally, a UV lamp with a wavelength range per the specifications outlined in MIL-STD-1330D is used. If any part of the three step inspection process reveals contamination, the part is cleaned again and re-inspected.

C. Installation or Storage of Cleaned Parts

Once cleaned, inspected, and dried the part is immediately installed or bagged and tagged for later use. Before a bagged part is installed into a system, SpaceDev repeats the inspection process to verify that the part was not contaminated while stored.

VI. Conclusion

Nitrous Oxide has properties which make it an ideal oxidizer for a variety of rocket and combustion applications. AMROC and SpaceDev have a long history of the safe use of nitrous oxide in hybrid rocket testing. While nitrous oxide is generally more stable and safer than other oxidizers used in rocketry, it still should always be handed only by personnel with the proper training and procedures.

As previously stated, this paper is intended only to highlight how SpaceDev handles nitrous oxide and is not intended to be used as a comprehensive guide on the design of nitrous oxide systems or nitrous oxide safety. While SpaceDev has done its best to verify that all the information contained in this paper is factually correct, SpaceDev assumes no liability for errors contained within this paper.

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