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TESTING AND CONTROL OF MATERIALS

USED IN HIGH PRESSURE OXYGEN CRYOGENIC SYSTEMS

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Introduction

Materials used in the crew bay, in systems interfacing with the crew bay, and in the propellant systems for the Apollo spacecraft are controlled by the Apollo Spacecraft Program Office document, "Apollo Spacecraft Nonmetallic Materials Requirements."(1) The Apollo spacecraft materials requirements for cryogenic oxygen systems are the same as those employed for gaseous oxygen systems (GOX). Tests have been conducted which show flammability testing conditions to be more severe with an atmosphere of ambient temperature GOX than an atmosphere of supercritical oxygen at the same test pressure or liquid (LOX) at ambient pressures.

Modifications were made in Addendum 2 of "Apollo Spacecraft Nonmetallic Materials Requirements" in revising Category D, materials used in greater than 20 psia oxygen, and in adding Category J, materials used in other hazardous fluids, such as propellants. The major revisions to Category D were in extending the existing nonmetallic materials requirements to metals which are normally exposed to the oxygen atmosphere and to all materials, metals and nonmetals, used in the oxygen systems which could be exposed to oxygen as a result of a single barrier failure.

Materials usage for each spacecraft are tracked by the COMAT (Characteristics of Materials) system. This computerized system relates the material to a specific component and lists the quantity used and the test results indicator for the material and/or component. The COMAT system functions to maintain configuration management for all controlled materials in the spacecraft.

Brief discussions of the materials and configurations testing techniques and requirements for the Apollo spacecraft high pressure oxygen systems are presented in the remainder of this paper.

Materials Testing Requirements For High Pressure Oxygen

Metallic Materials

Metallic materials used in high pressure oxygen are required to successfully complete the pneumatic impact test. Metallic materials which are used in dynamic applications such as valve seats are also required to successfully undergo mechanical impact testing.

Nonmetallic Materials

Nonmetallic materials used in high pressure oxygen are required to successfully complete the flash and fire point test, odor test, total organics and carbon monoxide tests, and pneumatic impact test. Nonmetallic materials which are used in dynamic applications also must successfully undergo the mechanical impact testing.

Materials Behind Potential Single Barrier Failures

As previously stated, these above testing requirements apply to materials which are normally exposed to oxygen, as well as those which would be exposed as a result of a single barrier failure. All components are evaluated for the probability of a single barrier failure which would allow oxygen into an enclosed compartment which is not normally a wetted area. The evaluation takes into account such considerations as leakage history, barrier thickness and design, acceptance testing, other spacecraft testing, and burst data. All materials in components which do not have a solid barrier; i.e., those having welded, brazed or mechanical joints, have been tested.

Materials Screening Tests

Only a brief discussion of the materials testing techniques will be presented here. All of the testing described is performed by the Manned Spacecraft Center's White Sands Test Facility and complete descriptions can be found in "Apollo Spacecraft Nonmetallic Materials Requirements."

High Pressure Flash and Fire Point Testing

High pressure flash and fire point testing is performed on all nonmetallic materials in their maximum systems usage pressure. The materials must have a minimum flash point of 400°F and a minimum fire point of 450°F.

Testing is accomplished by placing a half gram sample in the sample cup of the test cell, shown in Figure 1, and adjusting to the proper oxygen pressure. The sample cup is then heated at a rate of 25°F per minute while arcing above the sample for every 4 ± 1 second, 50 ± 20 millijoule spark. The above process is continual to 1000°F or until a flash and/or fire point of the material is obtained. A flash or a fire is detected by a photocell detector and fed into the storage oscilloscope. The high pressure flash and fire point apparatus has a present capability of 50 to 3000 psia. A similar system is employed for flash and fire point testing in pressures less than 50 psia.

Gaseous Oxygen Mechanical Impact Testing

GOX mechanical impact testing is performed on all materials at 1.5 times their maximum system usage pressure. The materials must show no evidence of reaction at that pressure to be acceptable for use in a dynamic application.

The mechanical impact testing apparatus utilizes a modified Army Ballistics Missile Agency impact tester as shown in Figure 2. The basic modifications include using a 7-1/2 pound plummet in lieu of the 20 pound plummet and replacing the anvil with a high-pressure test chamber as shown in Figure 3. All testing for the Apollo Spacecraft Program is done with a 50 foot-pounds per square inch impact which is on the order of six times greater than the highest impact load component used in the Apollo spacecraft.

A clean sample is placed in the sample cup and the system is purged and pressurized with oxygen to the specified test pressure. The pneumatic amplifier chamber is then pressurized with gaseous nitrogen to equalize the pressure on the striker pin. The plummet is then released and the impact energy is transmitted to the specimen through the striker pin. The specimen is then removed and inspected for evidence of a reaction. The mechanical impact system presently has a 7500 psia capability.

Gaseous Oxygen Pneumatic Impact Testing

GOX pneumatic impact testing is performed on all materials at 1.33 times their maximum system usage pressure. Materials must show no evidence of reaction to be acceptable for use in a high pressure oxygen system.

Testing is accomplished in the apparatus diagramed in Figures 4 and 5. A clean sample is placed in the sample cup and the cup is loosely installed into the system. The system is then purged with low pressure oxygen and the sample cup tightened. The system is monitored and pressure, temperature, and valve cycle times are recorded. Testing is accomplished with an automatic sequencer which provides identical pressure cycles. The sequencer first opens the high speed valve (2 milliseconds to full open) for 50 milliseconds. The valve then closes and holds the pressure for 5 seconds before venting down to atmospheric pressure. The pressure-vent sequence is repeated 4 more times per sample before removing and inspecting the sample for evidence of reaction. A high magnitude reaction will also be noted by the temperature readings.

High Pressure Autoignition Testing

High pressure autoignition testing was implemented to supplement the high pressure flash and fire point testing for materials used in higher than 3000 psia systems. Materials are tested at their maximum system usage pressure and must exhibit a minimum autoignition point of 450°F.

Testing is accomplished in an apparatus similar to the flash and fire point apparatus without the arcing equipment and with a thermocouple in lieu of a photocell for detecting a reaction. The sample and chamber preparation for this test is the same as for the flash and fire point testing. The sample is heated at the rate of 25°F per minute until the sample reaches 1000°F or autoignites. The autoignition system is capable of testing to 7500 psia.

Odor Testing

Materials are screened for undesirable odors by an odor panel of five to ten members. A sample equal to five grams per liter of test chamber is placed in the chamber. The chamber is then evacuated to 1 Torr or less and back filled with oxygen to approximately 5 psia. The sample and chamber are then heated inside an oven to 155°F for 72 hours. Next, the chamber is pressurized to one atmosphere with oxygen and sampled. Samples are then diluted with oxygen on a ratio of 29 parts of oxygen to 1 part of sample and 9 parts of oxygen to 1 part of sample. These two dilutions and an undiluted sample are then rated by the panel members on a scale of 0 to 4. A material which has an average rating on the undiluted sample of 2.5 or lower is considered acceptable.

Total Organics and Carbon Monoxide

A sample is prepared and heated for 72 hours as described for the odor testing. Gas samples are taken after the chamber reaches room temperature. The test specimen is then weighed and the gas samples analyzed for total organics and carbon monoxide. Total organics are expressed as pentane equivalents and shall not exceed 100 micrograms per gram of sample and carbon monoxide shall not exceed 25 micrograms per gram of sample for the material to be acceptable.

Test Results

Test results for the mechanical impact test show testing at the service module oxygen tank pressure of 1000 psia GOX to be more severe than testing at the same impact levels in ambient LOX. Tests are currently underway to compare the same materials at the same impact levels in a 1000 psia supercritical oxygen atmosphere.

Approximately 650 materials comprising over 6000 tests have been conducted for certification of the materials used in the current Apollo spacecraft high pressure oxygen systems. Of these tests, more than 90 percent were GOX pneumatic and mechanical impact or high pressure flash and fire point. Testing has indicated that no adverse flammability problem exists with the materials currently used in the Apollo spacecraft oxygen systems. All test data generated by these screening tests in support of the Apollo program are distributed to the Apollo spacecraft contractors in "Materials Test Data for Applications in High Pressure Oxygen and Other Hazardous Fluids," "Material Test Data by Generic Identification," and "Materials Test Data by Manufacturer's Designation." (2) (3) (4)

Configuration Flammability Testing Requirements

Configuration testing is required for all components that have materials associated with an ignition source or that are within one inch of an ignition source. Ignition sources may be either electrical or dynamic impact. Configuration testing may also be required for components which have materials that fail any of the required screening tests.

Configuration Flammability Tests

Configuration testing for the Apollo spacecraft oxygen systems is conducted on both the subassembly and assembly levels. Each configuration test is designed to meet the specific worst case operating conditions of the component. Specific examples of the configuration tests conducted for the service module cryogenics oxygen system are given below.

Subassembly Tests

A good example of a subassembly test is the testing which was conducted on the wiring that is used in the redesigned service module oxygen tank. Current overload and arcing tests were conducted in 1035 psia GOX on both faulted wires, which allow an oxygen path to the conductor, and unfaulted wires and with and without circuit protection.

Early overload testing through the spacecraft circuit protection and under the right conditions of a faulted wire to allow an oxygen path showed a need to enhance the design of the circuit protection with the addition of a 5 ampere fuse for the third heater element of the cryogenic oxygen tanks' heater probes. Faults in the sheath ranged in size from .004 inch to 1/16 of an inch in diameter with ignitions occurring in both extremes. The testing also showed that overloading an unfaulted wire to fusion resulted in the conductor losing continuity in many places while the sheath remained intact with no ignition occurring. Testing through the enhanced circuit protection showed that the cable could not be overloaded or ignited even when faulted. From these tests, it was concluded that ignition would require a multipoint failure mode consisting of a shorted wire, a breakdown of the circuit protection, and a fault to allow an oxygen path to the conductor.

Overload tests were also conducted on the wire in 1035 psia supercritical oxygen. Results from this test showed even a faulted wire without circuit protection would not ignite.

Assembly Tests

A series of tests was conducted on the Service Module oxygen tank heater probe. In one of the tests, a heater element was shorted approximately eight inches from the junction between the copper cold lead and the nichrome heater element. The shorted element had a fault to allow an oxygen path to the nichrome wire. The shorted element was protected by a 5.0 ampere fuse and a 5.0 ampere circuit breaker in series. The heater probe was preheated to various temperatures up to 400°F and a current of 14 amperes at 32 VDC was applied to the shorted element. In all of the tests, the fuse opened in a time ranging from 0.8 second to 2.2 seconds. The test was then repeated twice at 400°F using only the 5 ampere circuit breaker for circuit protection and each time the circuit breaker opened in 1.2 seconds at 14 amperes 32 VDC. Next, the test was repeated without a fuse or a circuit breaker. A current of 14 amperes at 32 VDC was maintained on the shorted wire and maximum steady temperature of 577°F was obtained without fusing or igniting the wire.

In the final test on the heater probe, a new short was made approximately three inches from the cold junction on the same heater element which was previously shorted. No circuit protection was provided. By applying 5.5 amperes to each of the heater elements, the probe was heated to 310°F. Amperage was then dropped to 2 amperes on the shorted wire and then increased to 16 amperes within one second. Current was increased to 17 amperes after 30 seconds. The heater element fused open at 17 amperes without causing ignition.

Other assembly tests were conducted on various Apollo spacecraft oxygen supply systems as required to verify their acceptability prior to Apollo 14.

Findings

Materials and configuration test data have indicated that the best metals available for high pressure oxygen systems are nickel and high nickel base alloys. Stainless steels while being harder to ignite than the softer metals such as aluminum still propagate when ignited. Data to date show that all nonmetallic materials, when supplied with enough energy, will ignite and propagate. All nonmetallic materials which were known to be directly exposed to the high pressure oxygen systems of the Apollo spacecraft have been removed or relocated so as not to be in direct contact with an electrical ignition source. As mentioned previously, test results to date show that an atmosphere of ambient temperature GOX is a more severe testing atmosphere than ambient pressure LOX or supercritical oxygen at the same test pressure as the GOX test pressure.

References

1. Anon.: Apollo Spacecraft Nonmetallic Materials Requirements, MSC-PA-D-67-13, Apollo Spacecraft Program Office, Manned Spacecraft Center.

2. Anon.: Materials Test Data for Applications in High Pressure Oxygen and Other Hazardous Fluids, NB/RT-71-45, Reliability Division, Manned Spacecraft Center.

3. Anon.: Material Test Data by Generic Identification, RPT-X52-32-914D, Reliability Division, Manned Spacecraft Center.

4. Anon.: Material Test Data by Manufacturers Designation, RPT-X52-32-914G, Reliability Division, Manned Spacecraft Center.

WSTF - FLASH AND FIRE POINT TEST APPARATUS AND CONTROL SYSTEM

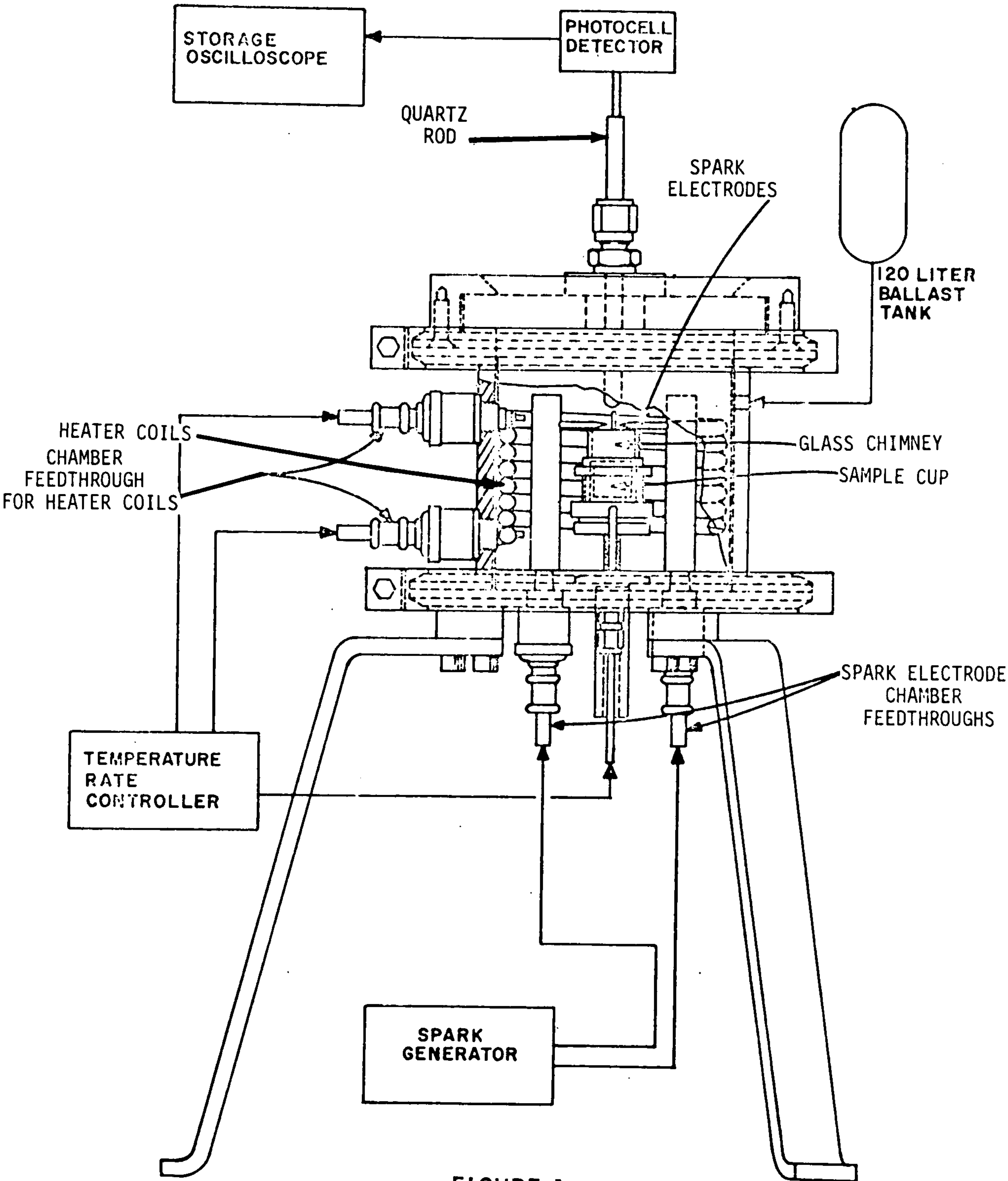

FIGURE 1

WSTF - MECHANICAL IMPACT TEST CHAMBER INSTALLED ON BASIC ABMA TESTER

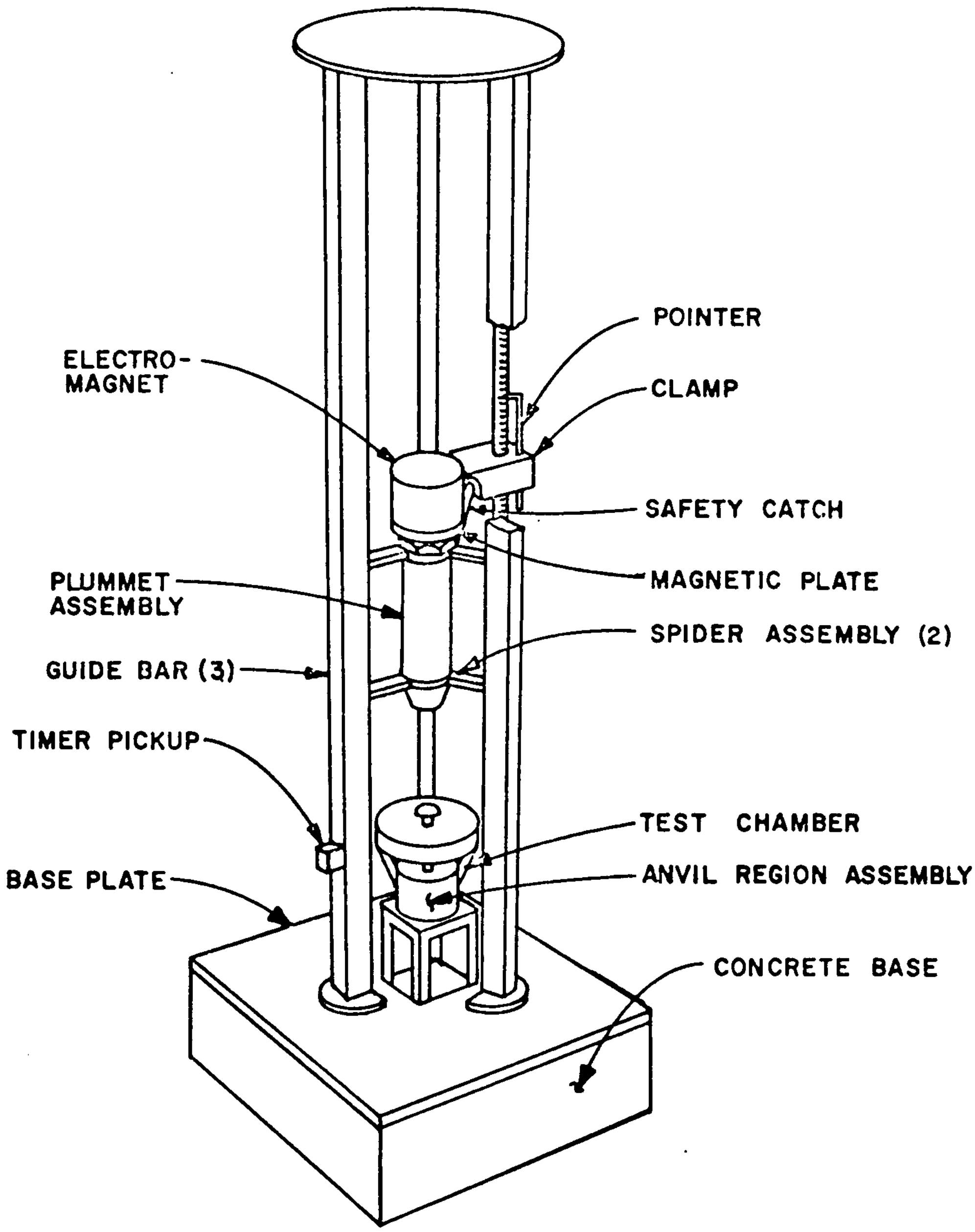

FIGURE 2

WSTF - MECHANICAL IMPACT TEST CHAMBER

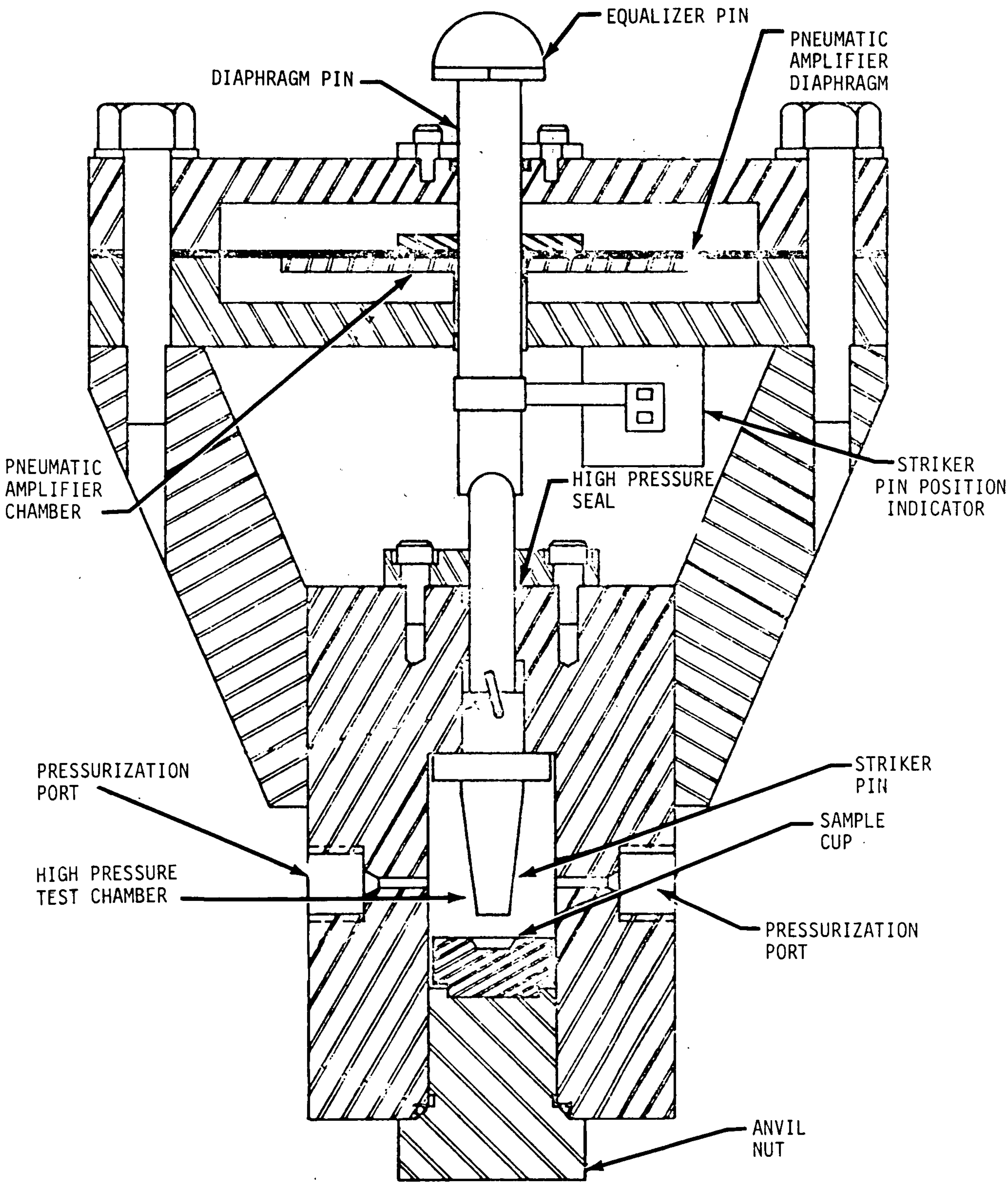

FIGURE 3

WSTF - PNEUMATIC IMPACT TEST SYSTEM

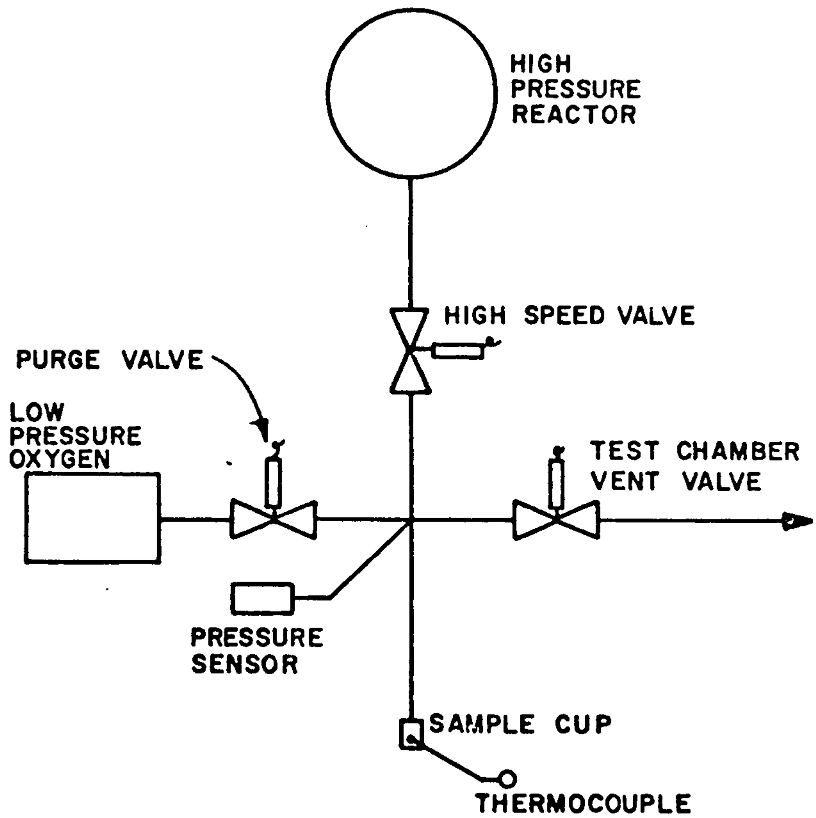

FIGURE 4

WSTF - PNEUMATIC IMPACT TEST SYSTEM

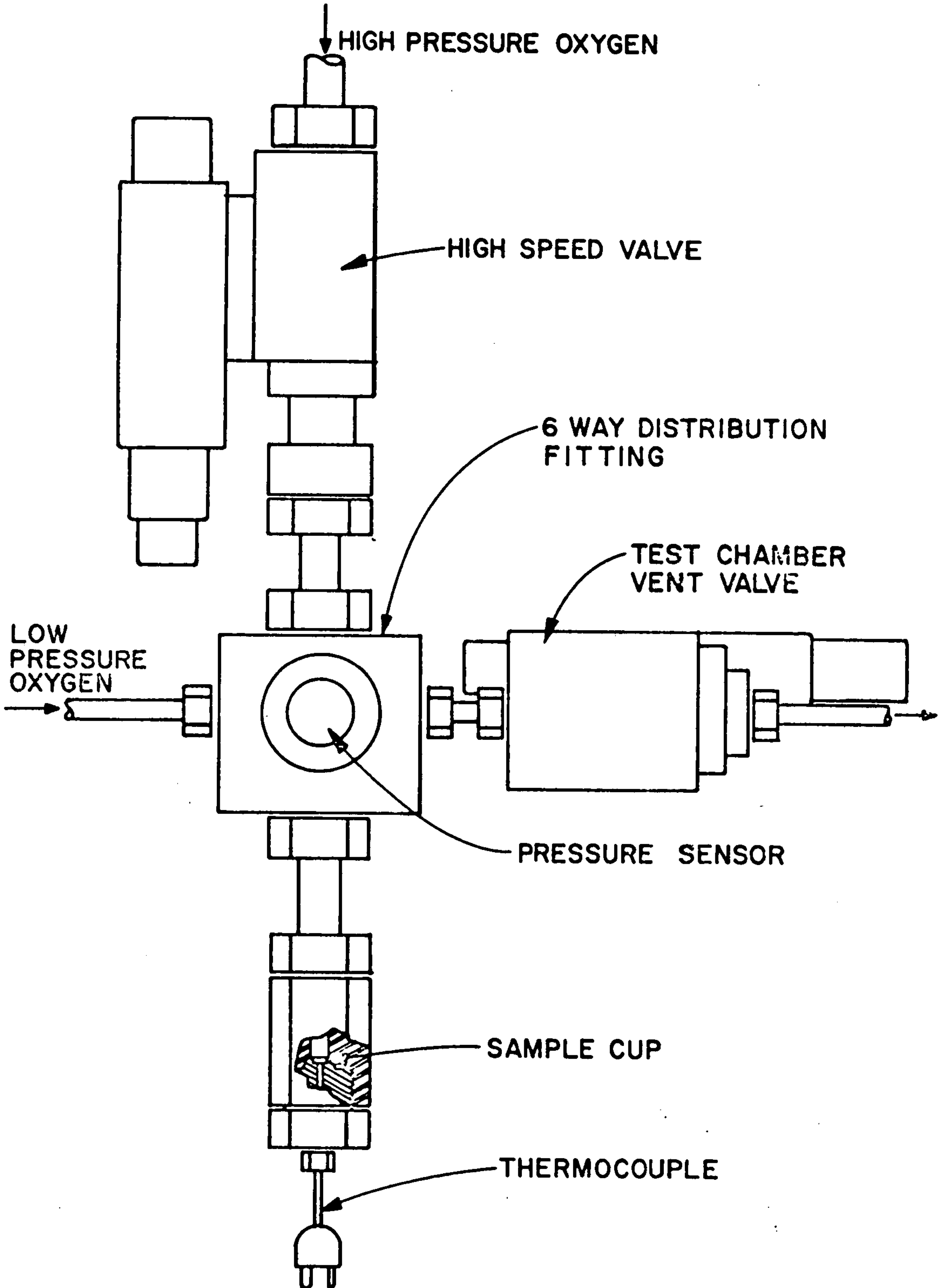

FIGURE 5