TEST METHODS FOR DETERMINING THE SUITABILITY OF METAL ALLOYS FOR USE IN OXYGEN-ENRICHED ENVIRONMENTS

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ABSTRACT

Materials are more flammable in oxygen-enriched environments than in air. When the structural elements of a system containing oxygen ignite and burn, the results are often catastrophic, causing loss of equipment and perhaps even human lives. Therefore, selection of the proper metallic and nonmetallic materials for use in oxygen systems is extremely important. While test methods for the selection of nonmetallic materials have been available for many years, test methods for the selection of metal alloys have not been available until recently. Several test methods are presented that were developed recently at NASA’s White Sands Test Facility (WSTF) to study the ignition and combustion of alloys, including the supersonic and subsonic speed particle impact tests, the frictional heating and coefficient-of-friction tests, and the promoted combustion test. These test methods are available for commercial use.

INTRODUCTION

Because nearly all metallic and non-metallic structural materials are flammable in oxygen-enriched atmospheres, costly fires have occurred in oxygen systems. For example, catastrophic oxygen-related fires have occurred in the space program, hindering mission success and costing human lives [1]. In the medical community, fires in operating rooms have occurred during surgery of the head and neck [2-5]. In industry, numerous fires have been reported in regulators and manual valves, causing severe system damage and human injury [6-8].

Furthermore, advancing technology is creating a demand for higher oxygen-use temperatures and pressures. NASA is investigating the use of liquid oxygen (LO2) and liquid hydrogen (LH2) as propellants for propulsion systems for a new generation of space-based orbital transfer vehicles (OTVs). The new design concept for the LH2/LO2 engines involves the use of gaseous oxygen (GO2) at 533 K (500 °F) and 34.5 MPa (5000 psi) to drive the turbine in the LO2 turbopump. Industry is considering the use of gas cylinders at up to 27.6 MPa (4000 psi). The use of oxygen at these higher temperatures and pressures causes a concern for engineers because it increases the risk of fire.

No absolute solution exists for controlling fire hazards in oxygen systems. However, the possibility of oxygen-related fires can be diminished with proper design practices and careful selection of materials. Guidance regarding proper design practices for oxygen systems and the selection of nonmetallic materials is available in the open literature [1,9-11]. Until recently, however, test methods and selection procedures for metals were not readily available.

The development of test methods for determining the suitability of metals in oxygen-enriched atmospheres began at NASA’s White Sands Test Facility (WSTF) in the late 1970’s. Several methods to determine the ignitability and combustion characteristics of metals have been developed. Ignition characteristics are determined using the particle impact, frictional heating, and coefficient-of-friction tests; and combustion characteristics are determined using the promoted combustion test [9,10]. This paper describes each of these test methods.
PARTICLE IMPACT TEST METHODS

Particle impact has been recognized as an ignition source in oxygen systems for several years [12-15]. Additionally, experience at WSTF has demonstrated that metal particles entrained in flowing oxygen can ignite valves, as seen in Figure 1. Two particle impact test methods, one operating at supersonic velocities and the other at subsonic velocities, have been developed at WSTF. The methods are used to determine the minimum temperature, pressure, and velocity at which particle impact ignition occurs.

Supersonic Particle Impact Test Method

The supersonic particle impact test chamber (Figure 2) has been described by Benz et al. [16]. It comprises a gas inlet and flow straightener, a particle injector, a converging nozzle, a diverging nozzle, and a test sample mounted on a holder. GO2 and the particle enter the inlet section and are accelerated to supersonic velocities as they pass through the converging and diverging nozzles. After flowing through a short constant-area section, the oxygen and the particle impact a sample made of the metal alloy being tested.

Typical supersonic impact of a 2000 μm- (0.08-in)-diameter aluminum particle results are shown in Figure 3. As the test sample temperature is increased, the susceptibility to ignition by particle impact increases.

Subsonic Particle Impact Test Method

The subsonic particle impact test chamber (Figure 4) has been described by Williams et al. [17]. It comprises a particle injector, a test sample, and a flow control orifice. Up to 5 g (0.01 lbs) of particles are injected into flowing oxygen and carried through the test chamber where they impact the test sample made from the metal alloy being tested. After impacting the sample, the oxygen and particles flow through holes on the sample periphery and are vented to the atmosphere through the flow control orifice.

Typical subsonic particle impact results are shown in Figure 5. For tests at ambient temperature with 2 g (0.004 lb) of iron powder, stainless-steel target ignitions do not occur until the particle velocity is increased to approximately 50 m/s (164 ft/s). When the temperature is increased to 360 to 450 K (188.6 to 350 °F), ignitions occur at 25 m/s (82 ft/s).
The supersonic and subsonic particle impact test methods are not patented, but are available for commercial use through NASA's technology transfer program.

**FRICIONAL HEATING TEST METHOD**

When mechanical components of a system rub together, heat is generated by friction. This frictionally generated heat has been identified as the cause of many fires in oxygen-enriched environments. For example, the rotating machinery shown in Figure 6 ignited and burned when a rotating part rubbed against the housing as the result of a bearing failure. Additionally, bearings in the space shuttle main engine high-pressure oxygen turbopump have ignited because of frictional heating when they failed during off-limit tests. The frictional heating test method was developed to determine metal alloys' susceptibility to frictional ignition in oxygen.

The frictional heating test apparatus shown in Figure 7 has been described by Benz and Stoltzfus [18]. It comprises an electrical drive motor and transmission assembly, a high-pressure test chamber, and a pneumatic cylinder. A rotating shaft extends through the test chamber and is connected at one end to the drive assembly.
Figure 6
Rotating Machinery Ignited by Friction

Test Gas Inlet/Vent Port
Protective Copper Sleeve
Test Cavity
Normal Force
Applied by
Pneumatic Cylinder
Thermocouples 1.3 mm and 5 mm from Rubbing Surface
Test Gas Thermocouple
Thermopile Port
Stationary Test Sample
Rotary Test Sample
Drive Motor and Transmission Assembly

Figure 7
Frictional Heating Test Apparatus
and at the other end to the pneumatic cylinder. The rotating test specimen is mounted on the shaft, and the stationary test specimen is affixed to the test chamber. Tests up to 27.6 MPa (4000 psi) in GO\textsubscript{2} and 2 MPa (300 psi) in LO\textsubscript{2} can be conducted in this test apparatus.

Typical results of frictional heating tests are shown in Table 1. The results are presented in terms of the product of the loading pressure (P) and the rubbing velocity (v) required for ignition of the material pair.

### Table 1

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>Stationary Rotary</th>
<th>Pv Product (W/m\textsuperscript{2} x 10\textsuperscript{-8})</th>
<th>Reaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inconel 718</td>
<td>Inconel 718</td>
<td>0.96-1.18 3.85</td>
<td>GOX Yes</td>
</tr>
<tr>
<td>Monel K-500</td>
<td>Monel K-500</td>
<td>1.37-1.69 4.15-4.23</td>
<td>LOX Yes</td>
</tr>
<tr>
<td>Inco MA754</td>
<td>Inco MA754</td>
<td>3.96-4.12 3.29-4.03</td>
<td>NO No</td>
</tr>
<tr>
<td>Haynes 214</td>
<td>Haynes 214</td>
<td>3.05 3.79-4.09</td>
<td>NO Yes</td>
</tr>
<tr>
<td>AMS 6278</td>
<td>AMS 6278</td>
<td>1.82 3.34</td>
<td>Yes Yes</td>
</tr>
<tr>
<td>Al 6061T6</td>
<td>Al 6061T6</td>
<td>0.48</td>
<td>NO ---</td>
</tr>
<tr>
<td>AI 2219</td>
<td>AI 2219</td>
<td>- 1.72</td>
<td>--- Yes</td>
</tr>
<tr>
<td>Monel K-500</td>
<td>Kel-F</td>
<td>- 0.23-0.45\textsuperscript{a}</td>
<td>--- No</td>
</tr>
<tr>
<td>Monel K-500</td>
<td>Vespel SP21</td>
<td>- 1.52</td>
<td>--- Yes</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Sample deformation before ignition

The LO\textsubscript{2} and GO\textsubscript{2} frictional heating test methods are patented and are available for licensing by NASA.

### COEFFICIENT-OF-FRICTION TEST METHOD

While frictional ignition data are available using the previously described test method, a need remains to understand the tribological behavior of metals in oxygen. A test apparatus is currently under development at WSTF for this purpose (see Figure 8).

The coefficient-of-friction test method comprises an electrical drive motor and transmission assembly, a pin-on-disk frictional contact device, and a high-pressure chamber. The drive motor and transmission assembly is similar to the one used in the frictional heating test. The pin-on-disk frictional contact device comprises a cylindrical test pin with a spherical end, a disk, two shafts that penetrate the chamber, and a load arm. The pin is mounted on the load arm, which is connected to the first shaft. A measured torque applied to this shaft by means of a pneumatic rotary actuator produces the normal contact force between the pin and the 5.1-cm-(2.0-in)-diameter test disk. The disk is mounted on the second shaft, which is connected to the drive motor assembly. As the pin contacts the rotating test disk, a frictional load is detected by a calibrated strain gauge mounted on the load arm. A 5.1-cm-(2.0-in)-diameter viewport in the chamber wall allows video coverage of the test. Tests at pressures up to 27.6 MPa (4000 psi) in GO\textsubscript{2} and 2 MPa (300 psi) in LO\textsubscript{2} can be conducted in this test system. Checkout tests are being conducted and a patent is pending on the coefficient-of-friction test method. It will be available for licensing once the patent is approved.

### PROMOTED COMBUSTION TEST METHOD

To determine if an alloy can be safely used in an oxygen system, its flammability must be known. If it is not flammable in a configuration similar to that in which it is intended for use, it can be used safely. The promoted
Combustion test measures flammability in terms of the minimum pressure required to support complete combustion of an alloy. This test method has been adopted by NASA as a standard for the selection of materials to be used in spacecraft. It is also being prepared as a standard by the American Society of Testing and Materials (ASTM) to determine the combustion behavior of metallic materials. The promoted combustion test apparatus shown in Figure 9 has been described by Stoltzfus et al. [19]. It comprises a cylindrical chamber, a copper liner and baseplate to protect the chamber from burning metal, and a sample mounting device. The chamber can be pressurized to 68.9 MPa (10,000 psia) and has four 5.1-cm- (2.0-in)-diameter viewports for sample observation and video recording. The test sample is held at the top in the sample mounting device and is ignited at the bottom by an aluminum or magnesium promoter. The promoter is ignited by an electrically heated wire.

Typical results of promoted combustion tests on several commonly used alloys are shown in Table 2. The table presents the minimum oxygen pressure required for complete combustion of 0.32-cm- (0.125-in)-diameter metal rods (threshold pressure). Nickel, copper, and their alloys are generally the least flammable. Iron-base alloys tend to be more flammable than the nickel- and copper-base alloys, but less flammable than the aluminum- and titanium-base alloys.

The promoted combustion test apparatus is patented and is available for licensing by NASA.
Table 2
Typical Promoted Combustion Test Results

<table>
<thead>
<tr>
<th>Material</th>
<th>Threshold Pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MPa (psia)</td>
</tr>
<tr>
<td>Monel K-500</td>
<td>68.9 &gt; 10,000</td>
</tr>
<tr>
<td>Inconel MA754</td>
<td>68.9 &gt; 10,000</td>
</tr>
<tr>
<td>Haynes 214</td>
<td>68.9 &gt; 10,000</td>
</tr>
<tr>
<td>Monel 400</td>
<td>68.9 &gt; 10,000</td>
</tr>
<tr>
<td>Brass 360 CDA</td>
<td>68.9 &gt; 10,000</td>
</tr>
<tr>
<td>Nickel 200</td>
<td>55.2 &gt; 8,000</td>
</tr>
<tr>
<td>Inconel 600</td>
<td>20.7 3,000</td>
</tr>
<tr>
<td>Inconel 625</td>
<td>20.7 3,000</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>6.9   1,000</td>
</tr>
<tr>
<td>304 SS</td>
<td>6.9   1,000</td>
</tr>
<tr>
<td>Aluminum 6061</td>
<td>0.68 100</td>
</tr>
<tr>
<td>Aluminum 99.9 %</td>
<td>0.17   25</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>0.007 1</td>
</tr>
</tbody>
</table>

APPLICATION OF TEST METHODS
Johnson Space Center, Kennedy Space Center, Marshall Space Flight Center, and Lewis Research Center have used all these test methods to evaluate metals for use in oxygen systems. The results have been used to select metal alloys for the shuttle main propulsion system (MPS) oxygen flow control valves, the shuttle MPS high pressure oxygen turbopump bearings, advanced propulsion systems for the Space Exploration Initiative (SEI), and ground support equipment. The Langley Research Center has used particle impact data to select materials to use in the transpiration-cooled nozzle design for a wind tunnel.

The Department of Defense has used the test methods to determine the relative compatibility of aluminum alloys that may be used in cryogenic propellant tankage in future advanced launch systems. The data obtained have also been used to determine ignition and combustion hazards on aircraft main, backup, and emergency oxygen supply systems. Finally, the ground carts that are used to resupply aircraft oxygen systems on the flight line are being evaluated and new designs are being considered, based on the data from these test methods.

ASTM sponsored a test program that used the particle impact, frictional heating, and promoted combustion test methods to test alloys used in industrial oxygen systems. ASTM has published over 21 papers in their Standard Technical Publications containing data from these test methods [20-24]. ASTM has also prepared a training course entitled “Controlling Fire Hazards in Oxygen Systems” that makes extensive use of data from these methods. Additionally, several commercial companies have used these test methods to make material selections for applications ranging from aircraft turbine engines to compressor seal design.

These test methods are not only used by engineers for designing safe oxygen systems but they are also used for scientific research in the field of metals combustion. They have been used as tools for understanding the theories of ignition and combustion of metals and alloys [25-27]. It is anticipated that this understanding will aid in the development of new burn-resistant structural alloys for use in oxygen environments.

In summary, fire hazard in oxygen systems is a serious problem. However, a solution that minimizes this hazard exists using data from the test methods described in this paper. These methods have been used extensively in the past and are available for commercial use in the future.
REFERENCES


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