APTech Test Report # 30011500 Revision 0

RAPID OXYGEN PRESSURIZATION OF AP3625 VALVES WITH 1/2 INCH DIAMETER UPSTREAM TUBING

June 4, 2004

Scope: This report summarizes testing in which APTech AP3625 valves were placed downstream of a 4 foot long, 1/2 inch outer diameter tube, and rapidly pressurized on the inlet port with oxygen, from atmospheric pressure to full cylinder pressure, 10 times in succession.

Copyright © June 2004
Advanced Pressure Technology
687 Technology Way
Napa, CA 94558
Telephone: 707-259-0102

CONFIDENTIAL

This test report is the property of Advanced Pressure Technology ("APTech") and may not be reproduced in whole or part without the written permission of APTech

APTech Document # 30011500 Rev. 0

1. Introduction

Rapid pressurization of a length of tubing from one end will result in heating of the gas at the far end of the tubing due to adiabatic compression of the low pressure gas originally in the tubing. It is suspected that such heating due to adiabatic compression can lead to the ignition of seats and other internal parts in oxygen gas system components if the system upstream of the component is pressurized too rapidly with high pressure oxygen.

Testing was performed to determine whether an AP3625 valve would ignite if placed in a gas test system immediately downstream of an approximately 4 foot (1.3 m) long, 1/2 inch (12.7 mm) outer diameter, section of tubing, and subjected to 10 cycles of rapid pressurization with oxygen from atmospheric pressure to cylinder pressure.

A total of four adiabatic compression cycle tests were performed. Two were performed using closed AP3625 valves without seat leaks and two were performed using AP3625 valves that leaked across the seat.

Previous adiabatic compression testing with oxygen by APTech using an approximately 3 foot (1 m) long section of 1/4 inch (6.3 mm) outer diameter tubing, was unsuccessful in obtaining ignition in either an AP1510 regulator or an AP3625 valve. Thus, in this testing, APTech significantly increased the volume of the upstream tubing to increase the volume of hot gas compressed into the far end of the tubing, and effectively transfer a larger amount of heat into the test components. This was an attempt to increase the likelihood of valve seat ignition.

In addition, previous adiabatic compression testing by APTech with nitrogen showed that significant melting and erosion of a valve seat could be obtained by creating a small seat leak prior to exposing the valve to rapid pressurization. The seat leaks in the nitrogen testing were created either by cracking open the valve slightly or by placing a small notch across the sealing surface of the seat, before exposing the valves to rapid pressurization. Based upon this information, it was hypothesized that a valve with a small seat leak would have a greater probability of igniting the seat material in an adiabatic compression test in oxygen compared to a valve without a seat leak.

2. Test Description

2.1 Components Tested

Standard production APTech AP3625 valves were used for the testing. The valves had PCTFE seats and were designed and manufactured for high purity semiconductor manufacturing applications. The wetted surfaces of the internal parts were burr free with 10 Ra or better surfaces finishes. The valve bodies were made from 316L VAR material and electropolished. All wetted parts were thoroughly degreased and cleaned prior to assembly using APTech's ultra-high purity, hot and cold de-ionized water, ultrasonic cleaning process, according to APTech documents DII-100 and DII-101. This process results in cleanliness levels exceeding those specified in document CGA G-4.1, "Cleaning Equipment for Oxygen Service". All test components were assembled in APTech's cleanroom to prevent particle contamination.

2.2 Test Setup

The test setup is shown in **Figure 1**. The setup was used to cycle the pressure P₁, upstream of the test component, with oxygen between atmospheric pressure and full cylinder pressure. The 1/2 inch (12.7 cm) outer diameter tubing upstream of the test component had a length of approximately 53 inches (134 cm) and an inner diameter of .40 inches (10.2 mm). All test setup components were thoroughly degreased and cleaned prior to assembly using APTech's standard cleanroom hot and cold de-ionized water,

APTech Document # 30011500 Rev. 0

ultrasonic cleaning process, according to APTech documents DII-100 and DII-101. Nickel gaskets were used for all face seal connections. 90 psig (6.1 barg) of nitrogen gas regulated from a bulk nitrogen supply was used to actuate the pneumatic valves during the testing.

A 3000 psig (207 barg) pressure transducer was used to read pressure P_1 remotely. The pressure transducer was attached to a data acquisition system to record the fill time required to pressurize the section of tubing upstream of the test component. The data acquisition system recorded 240 samples per second.

Rapid Oxygen Pressurization of AP3625 Valves with 1/2 Inch Diameter Upstream Tubing APTech Document # 30011500 Rev. 0

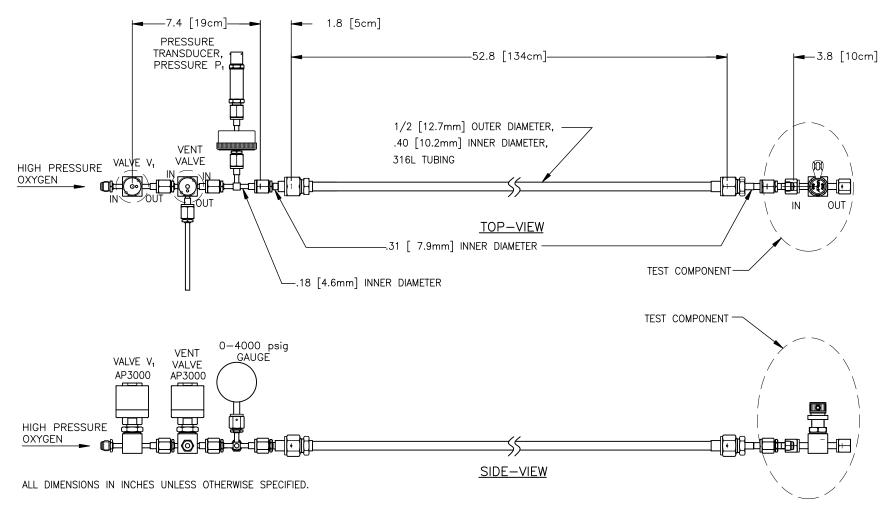


Figure 1. Test setup.

2.3 Test Procedure

Four separate adiabatic compression cycle tests were run. In the first two tests, designated as tests 1 and 2 in this report, the test valves were closed with no flow across the seat prior to being subjected to the rapid pressurization on the inlet port.

For the third and fourth tests, designated as tests 3 and 4, a small notch was created across the seat prior to pressurization. The notch was created as follows. The AP3625 valves were disassembled and a .006 inch (.152 mm) diameter, stainless steel wire was placed across the seat. The valves were reassembled with the wire in place and the actuator knobs closed with 25 in·lbf (2.82 N·m) to press the wire into the sealing surface of the seat. The valves were disassembled again and the wire removed, leaving a small notch across the sealing surface of the seat. The valves were reassembled again.

In test 3, the valve was pressurized in the closed position. The flow rate across the seat due to the notch, was less than 1 slpm with 2000 psig (138 barg) of nitrogen on the inlet.

In test 4, the valve was cracked open slightly to achieve approximately 5 slpm of flow with 2000 psig (138 barg) of nitrogen on the inlet.

2.4 Pressurization Procedure

An AP3625 valve was installed into the test setup as shown in Figure 1.

The internal volume between valve V_1 and the test component, was pressurized from atmospheric pressure to full cylinder pressure (approximately 2000 psig, 138 barg) of oxygen, 10 times in succession as follows:

- 1. High pressure oxygen at cylinder pressure was applied to the inlet port of valve V_1 with valve V_1 closed and the vent valve open.
- The vent valve was closed.
- 3. The data acquisition system was set to begin recording the pressure P₁ versus time.
- 4. From a remote location, valve V₁ was rapidly opened while pressure P₁ was monitored. 2 to 3 seconds later, valve V₁ was closed again.
- 5. The vent valve was opened for a few seconds to vent the line. Then the vent valve was closed again.
- 6. This process was repeated 9 times beginning at step 4. Each pressurization cycle was completed in approximately 5 to 10 seconds.

3. Test Results

The recorded fill time and maximum pressure for each pressurization cycle for all four tests are shown in Tables 1 through 4.

APTech Document # 30011500 Rev. 0

Table 1. Test data for test #1 (valve closed).

	Fill Time	Maximum Pressure P ₁	
Cycle	(seconds)	(psig) (barg)	
1	0.250	2000	138
2	0.250	1990	137
3	0.250	1980	137
4	0.241	1970	136
5	0.245	1970	136
6	0.250	1960	135
7	0.241	1950	134
8	0.259	1950	134
9	0.288	1940	134
10	0.271	1930	133

Table 2. Test data for test #2 (valve closed).

	Fill Time	Maximum Pressure P ₁	
Cycle	(seconds)	(psig) (barg)	
1	0.250	1960	135
2	0.263	1950	134
3	0.287	1940	134
4	0.279	1930	133
5	0.283	1920	132
6	0.283	1920	132
7	0.279	1910	132
8	0.275	1900	131
9	0.283	1890	130
10	0.279	1890	130

APTech Document # 30011500 Rev. 0

	Table	3.	Test data	for test #3	(notched	seat.	valve closed
--	-------	----	-----------	-------------	----------	-------	--------------

	Fill Time	Maximum Pressure P ₁	
Cycle	(seconds)	(psig) (barg)	
1	0.241	2020	139
2	0.287	2020	139
3	0.283	2010	139
4	0.283	2000	138
5	0.283	1990	137
6	0.279	1980	137
7	0.292	1980	137
8	0.283	1970	136
9	0.283	1960	135
10	0.283	1960	135

Table 4. Test data for test #4 (notched seat, valve cracked open).

	Fill Time	Maximum Pressure P ₁	
Cycle	(seconds)	(psig)	(barg)
1	0.279	1970	136
2	0.283	1960	135
3	0.287	1950	134

No seat leaks or other signs of seat failure were observed during the pressurization cycles for tests 1 and 2. For test 3, the sound amplitude of the flow exiting the valve seemed to decrease significantly after the first couple of pressurization cycles. This indicated that the flow across the seat had decreased. During test 4, a large seat leak developed on the 3rd pressurization.

After the testing, the test components were disassembled and the internal parts inspected under a microscope. There were no indications of ignition on the seats or elsewhere inside any of the tested valves. The internal parts of the valves from tests 1 and 2 had no damage after the testing.

In the valve from test 3, the seat material around the notch appeared as though it had melted slightly and flowed into the notch. This could explain why the leak rate through the valve sounded like it decreased after the valve was exposed to the adiabatic compression cycles.

In the valve from test 4, a large channel had opened up across the sealing surface of the seat where the notch was previously. A photograph of the seat after the testing is shown in **Figure 2**. The channel was several times larger than the original notch. The seat material at the entrance to the channel on the inlet side had rounded edges and a glossy surface texture. On the outlet side of the channel, the PCTFE material was shredded into numerous, fine, hair like stringers. It appeared that the hot gas from the adiabatic compression had melted the surface of the material around the notch allowing the high pressure gas to blow open a larger opening through the seat. It did not appear that any of

APTech Document # 30011500 Rev. 0

the seat material had ignited. The damaged seat appeared very similar to a valve seat that APTech had tested under similar conditions in nitrogen.



Figure 2. Valve seat from test 4 after rapid pressurization with high pressure oxygen.

4. Conclusion

Four tests were run in which an AP3625 valve was placed in a gas test system downstream of an approximately 4 foot (1.3 m) long, 1/2 inch (12.7 mm) outer diameter, section of tubing, and subjected to 10 cycles of rapid pressurization with oxygen from atmospheric pressure to approximately 2000 psig (138 barg) of cylinder pressure. There were no signs of ignition on the internal parts of any of the four test valves after the testing.

The first two tests were performed using valves without leaks across the seat. The adiabatic compression cycles did not damage the seats of the valves from these two tests.

The third test was performed with a closed valve that had a small seat leak due to a small notch placed across the sealing surface. In this valve, the heat from the adiabatic compression cycling caused the material around the notch to yield and flow into the notch. This appeared to cause the leak rate across the seat to drop.

The fourth test was performed with a valve that had a larger seat leak due to it having a small notch across the seat and the valve being cracked slightly open. After the adiabatic compression cycles, the seat of this valve was severely damaged. A large channel had blow through the sealing surface of the seat. It appeared that the seat material around the notch had melted and then blown through to the low pressure side of the valve.

Although we were able to get the seat material to melt in the last two tests, there were no signs of ignition on the seats or any other internal parts of the valves in any of the tests. The seats of the valves we tested in oxygen looked very similar to seats that were exposed to adiabatic compression testing in nitrogen.

Report by:

Jim Moser Project Engineer