Test Report # 30016497 Rev 2

Nitrous Oxide Compatibility Test

Scope: This test report contains chemical compatibility test data of nitrous oxide with PCTFE (Daikin Neoflon M-400H), polyimide (Dupont Vespel SP-1), and PTFE (DuPont Teflon grade 8A).

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Introduction

Historical review

When nitrous oxide (N_2O) gained popularity as a semiconductor process gas in the early 1990s, sporadic failures occurred in semiconductor valves and pressure regulators with PCTFE seats. Typically, isolation valves shut off or restricted flow in the open position due to seat swelling and pressure regulators developed across-the-seat leaks due to seat material yielding. Valves that shut off or restricted flow were spring type diaphragm valves that contained relatively large volumes of PCTFE material. Regulators that developed seat leak were tied diaphragm type with limited structural support above the seat. Failed valves and regulators were located in the gas source manifold and exposed to full cylinder pressure.

Polyimide (e.g DuPont Vespel® SP-1) was proposed as a seat material to solve the problems. After some initial success, it became common practice to specify "Vespel" for valve and regulators used in nitrous oxide service in the semiconductor industry. Although failures had been limited to the source gas manifold, the use of "Vespel" seats became standard throughout the process gas line components from the gas source manifold to point-of-use.

The use of polyimide has eliminated nitrous oxide compatibility failures at semiconductor facilities, but at a cost. Polyimide material is more expensive than PCTFE and some other seat materials. In addition, polyimide has a very high compressive strength, which requires a high load to form a good seal and reduces the seal width, making the component more susceptible to seat leaks. This can be a problem in low pressure pneumatic valves (such as the AP3540 or AP4540) that have lower closing loads and in mini pressure regulators (such as the AP500) which have small diaphragms and low poppet spring forces. For these reasons, component suppliers increase the price of valves and regulators with polyimide seats.

Test Parts

It was proposed to evaluate the effect of nitrous oxide on PCTFE, polyimide, and PTFE under low pressure point-of-use conditions (150 psig or less) and source gas manifold pressures (nitrous oxide vapor pressure of 745 psig at 68 degrees F). Due to high flow conditions, it is possible that liquid nitrous oxide can form, so it was also proposed to evaluate exposure to liquid nitrous oxide.

This test report evaluates samples of PCTFE (Daikin Neoflon™ M400-H), polyimide (DuPont Vespel SP-1), and PTFE (DuPont Teflon® grade 8A) exposed to nitrous oxide for swelling and compressive strength changes. The material samples were disc shaped—approximately 0.10 inch thick, 0.26 inch diameter, and 0.07 inch thru diameter hole.

Revision History

Rev 1: Removed note regarding product performance and specifications because the note is not applicable to this test report.

Rev 2: Corrected typographical error in Table 2.

<u>Trademark Information</u>

Neoflon is a trademark of Daikin Industries.

Teflon and Vespel are trademarks of DuPont.

Swell Test

Test Setup and Protocol

The thickness of the PCTFE, polyimide, and PTFE samples was measured. The samples were exposed to 100% nitrous oxide at three conditions: (1) pressurized to 150 psig, (2) pressurized to 745 psig, and (3) submerged in liquid nitrous oxide at 745 psig (vapor pressure). After 100 hours, the samples were removed from the nitrous oxide environment and immediately the thickness was again measured. All tests were done at room temperature conditions (68 degrees F).

The measurement accuracy was ±0.00010 inch and repeatability was ±0.00015 inch for a total measurement error of ±0.00025 inch or approximately ±0.25% of the sample thickness.

Results

The measurement results for the 150 psig exposure are shown in Table 1, for the 745 psig exposure are shown in Table 2, and for the 745 psig liquid exposure are shown in Table 3. The very slight swelling at 150 psig is right at the measurable range. The data at 745 psig in both gaseous and liquid exposures indicates that all materials had some swelling. The PCTFE increased ~5.5%, the polyimide increased ~1.5%, and the PTFE increased ~2.3%

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#1	PCTFE	0.09960	0.09985	0.25%	0.25%
#2	PCTFE	0.09970	0.09995	0.25%	
#7	Polyimide	0.10070	0.10100	0.30%	0.27%
#8	Polyimide	0.10055	0.10080	0.25%	
#13	PTFE	0.09945	0.10000	0.55%	0.48%
#14	PTFE	0.09950	0.09990	0.40%	

Table 1. Sample Measurements – 150 psig Exposure

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#3	PCTFE	0.09980	0.10355	3.76%	5.09%
#4	PCTFE	0.09960	0.10600	6.43%	
#9	Polyimide	0.10060	0.10210	1.49%	1.47%
#10	Polyimide	0.10070	0.10215	1.44%	
#15	PTFE	0.09960	0.10185	2.26%	2.19%
#16	PTFE	0.09945	0.10155	2.11%	

Table 2. Measurements – 745 psig Exposure

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#5	PCTFE	0.09975	0.10555	5.81%	5.77%
#6	PCTFE	0.09950	0.10520	5.73%	
#11	Polyimide	0.10060	0.10215	1.54%	1.42%
#12	Polyimide	0.10075	0.10205	1.29%	
#17	PTFE	0.09950	0.10210	2.61%	2.39%
#18	PTFE	0.09965	0.10180	2.16%	

Table 3. Measurements – 745 psig Liquid Exposure

Compression Test

Test Setup and Protocol

The thickness of PCTFE, polyimide, and PTFE samples was measured. The samples were exposed to 100% nitrous oxide at three conditions: (1) pressurized to 150 psig, (2) pressurized to 745 psig, and (3) submerged in liquid nitrous oxide at 745 psig (vapor pressure). After 100 hours, the samples were removed from the nitrous oxide environment, and immediately the thickness was measured and the seat was placed in a spring tester and a load was applied to obtain deflections of 0.001, 0.005 and 0.010 inch (approximately 1, 5, and 10% of material thickness). The load at each deflection point was recorded. After the load test, the thickness was again measured. All tests were done at room temperature conditions (68 degrees F).

The thickness measurement accuracy was ±0.00010 inch and repeatability was ±0.00015 inch for a total measurement error of ±0.00025 inch or approximately ±0.25% of the thickness. The load measurement accuracy was ±1 lbf and the deflection measurement accuracy was ±0.001 inch. Due to the deflection measurement accuracy, the data point at 0.001 inch for load has significant associated error.

Results

The results of the load versus deflection test for the PCTFE material are shown in Figure 1, for the polyimide material are shown in Figure 2, and for the PTFE material are shown in Figure 3. The data indicates that no significant change in compressive strength occurs with 150 psig exposure, but that the PCTFE material has a significant change and the PTFE has a slight change when exposed to 745 psig gaseous and 745 psig liquid nitrous oxide. The thickness change after exposure to nitrous oxide and before load testing is consistent with the first swell test.

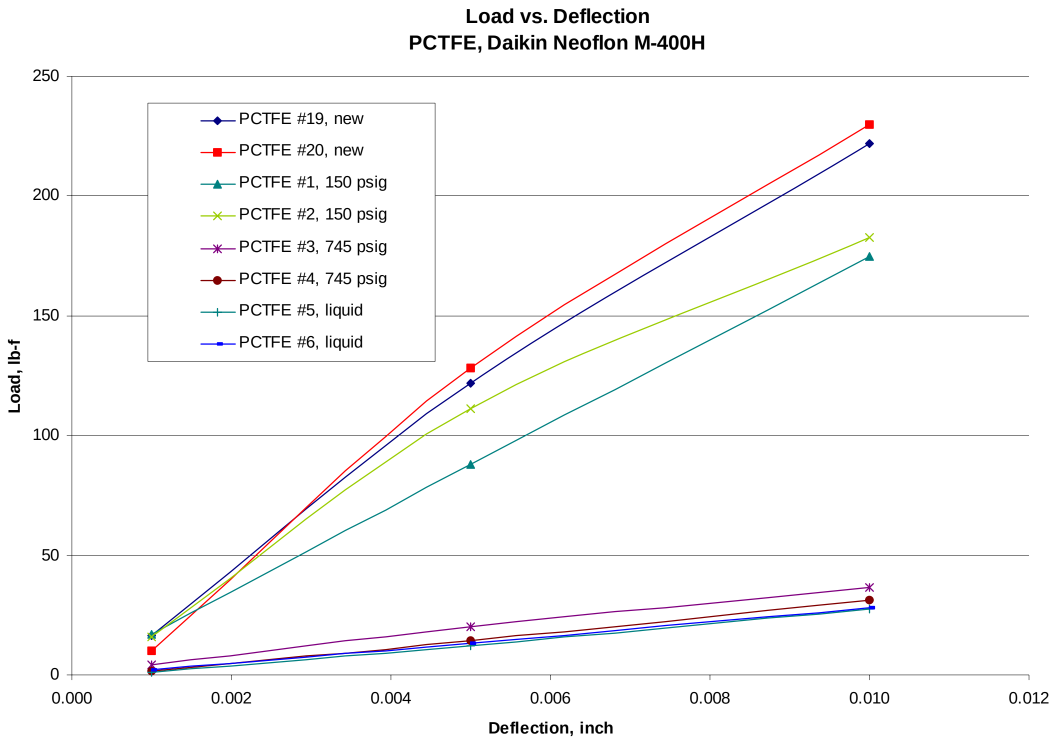

Figure 1. PCTFE Load vs. Deflection Results

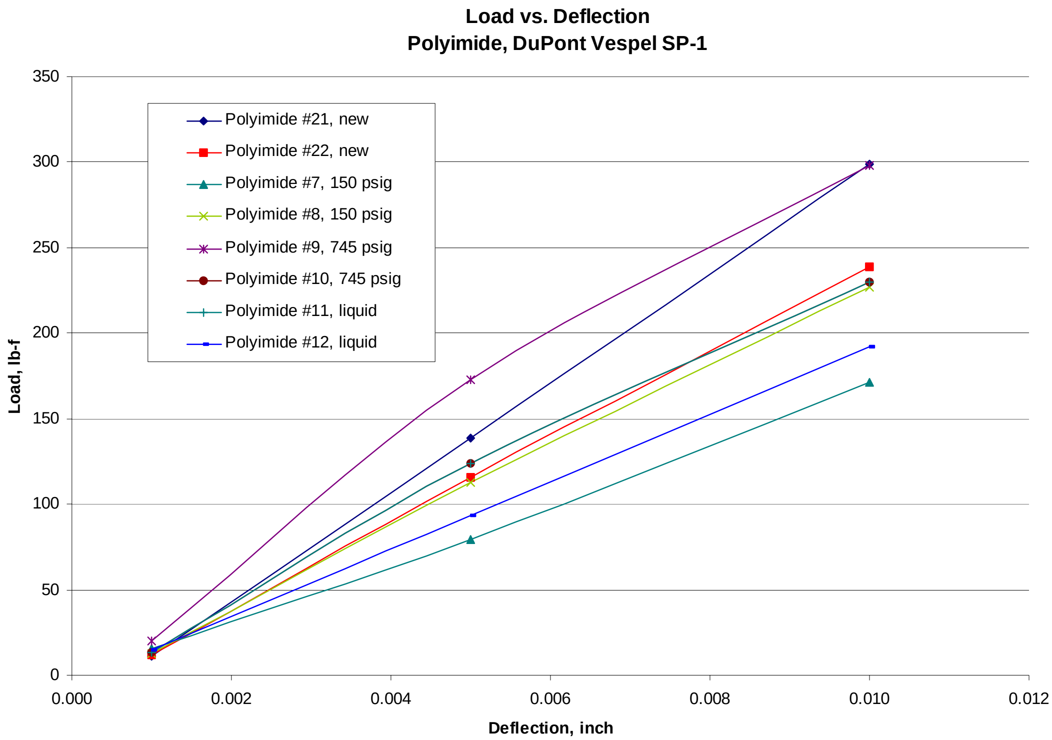

Figure 2. Polyimide Load vs. Deflection Results

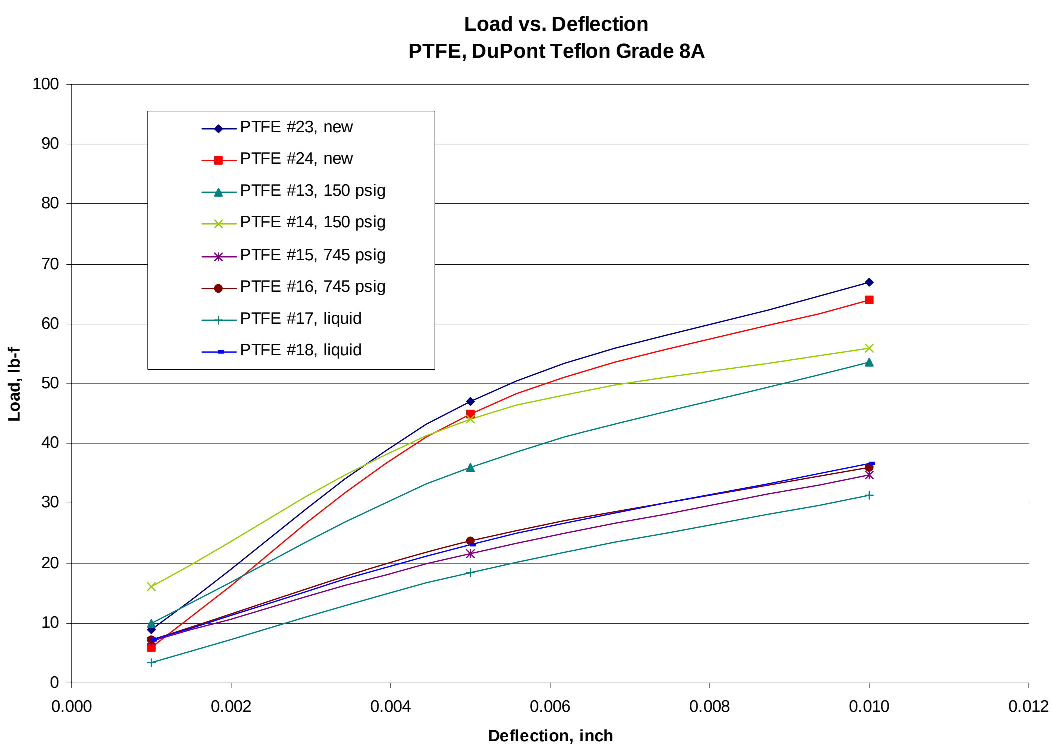

Figure 3. PTFE Load vs. Deflection Results

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#1	PCTFE	0.09960	0.09980	0.20%	0.23%
#2	PCTFE	0.09970	0.09995	0.25%	
#7	Polyimide	0.10070	0.10095	0.25%	0.25%
#8	Polyimide	0.10055	0.10080	0.25%	
#13	PTFE	0.09945	0.09990	0.45%	0.30%
#14	PTFE	0.09950	0.09965	0.15%	

Table 4. Sample Measurements – 150 psig Exposure

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#1	PCTFE	0.09980	0.09955	-0.25%	-0.35%
#2	PCTFE	0.09995	0.09950	-0.45%	
#7	Polyimide	0.10095	0.10090	-0.05%	-0.05%
#8	Polyimide	0.10080	0.10075	-0.05%	
#13	PTFE	0.09990	0.09780	-2.10%	-2.21%
#14	PTFE	0.09965	0.09735	-2.31%	

Table 5. Thickness Change after Load Test, 150 psig Exposure

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#3	PCTFE	0.09980	0.10355	3.76%	4.61%
#4	PCTFE	0.09960	0.10505	5.47%	
#9	Polyimide	0.10060	0.10250	1.89%	1.81%
#10	Polyimide	0.10070	0.10245	1.74%	
#15	PTFE	0.09960	0.10185	2.26%	2.03%
#16	PTFE	0.09945	0.10125	1.81%	

Table 6. Measurements – 745 psig Exposure

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#3	PCTFE	0.10355	0.10200	-1.50%	-1.30%
#4	PCTFE	0.10505	0.10390	-1.09%	
#9	Polyimide	0.10250	0.10160	-0.88%	-0.63%
#10	Polyimide	0.10245	0.10205	-0.39%	
#15	PTFE	0.10185	0.10030	-1.52%	-1.43%
#16	PTFE	0.10125	0.09990	-1.33%	

Table 7. Thickness Change after Load Test, 745 psig Exposure

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#5	PCTFE	0.09975	0.10570	5.96%	5.97%
#6	PCTFE	0.09950	0.10545	5.98%	
#11	Polyimide	0.10060	0.10250	1.89%	1.81%
#12	Polyimide	0.10075	0.10250	1.74%	
#17	PTFE	0.09950	0.10240	2.91%	2.41%
#18	PTFE	0.09965	0.10155	1.91%	

Table 8 745 psig Liquid Exposure

Test Sample	Material	Measurement, inch		Change	Average Change
		Before	After		
#5	PCTFE	0.10570	0.10390	-1.70%	-1.73%
#6	PCTFE	0.10545	0.10360	-1.75%	
#11	Polyimide	0.10250	0.10215	-0.34%	-0.32%
#12	Polyimide	0.10250	0.10220	-0.29%	
#17	PTFE	0.10240	0.10075	-1.61%	-1.62%
#18	PTFE	0.10155	0.09990	-1.62%	

Table 9. Thickness Change after Load Test, 745 psig Liquid Exposure

Summary

Samples of PCTFE (Daikin Neoflon M-400H), polyimide (DuPont Vespel SP-1), and PTFE (DuPont Teflon grade 8A) were exposed to gaseous nitrous oxide at 150 psig, gasious nitrous oxide at 745 psig, and liquid nitrous oxide at 745 psig. A swell test and compressive strength test was performed.

Swell test: The test data found very minor increase in sample thickness for all materials at 150 psig. The test data found that the thickness of all material samples increased at 745 psig—the PCTFE increased ~5.5%, the polyimide increased ~1.5%, and the PTFE increased ~2.3%. The test data found that 745 psig gaseous and 745 psig liquid exposure had virtually the same effect on the material thickness.

Compression test: The test data found a minor reduction in compressive strength for the PCTFE and PTFE samples exposed to 150 psig gaseous nitrous oxide. The test data found significant change in compression strength for the PCTFE samples exposed to 745 psig gasous and liquid nitrous oxide. The test data found a reduction in compressive strength for the PTFE samples exposed to 745 psig gasous and liquid nitrous oxide, but not to the same extent as the PCTFE samples. The test data indicated a minor reduction in compressive strength for polyimide exposed to nitrous oxide, but there was no obvious correlation to the individual exposure conditions.

The data in this report found that nitrous oxide has a very minor effect on the physical properties of PCTFE, polyimide, and PTFE at 150 psig. The data found that PCTFE and to a lesser extent PTFE will swell and have reduced compressive strength when exposed to 745 psig gaseous or liquid nitrous oxide. After exposure to 745 psig nitrous oxide, the PTCFE compressive strength dropped below that of PTFE, even though PCTFE samples not exposed to nitrous oxide had three times the compressive strength of PTFE. The data indicates that polyimide is the superior material for use at high pressure.

The data in this report supports the historical review on page 3. Swelling of PCTFE at high pressure explains why spring type diaphragm valves with short stroke (approximately 0.020 inch) and relatively large volumes of PCTFE material could restrict or shut off flow. The significant reduction in strength of PCTFE at high pressure explains why regulators developed across-the-seat leaks due to yielding of the seat material. No significant swelling or compressive strength change at 150 psig explains why failures were restricted to the gas source manifold and no failures were reported in valve manifold boxes (VMBs) or process tools.

Based on the data in this report, it is recommend that polyimide (Vespel SP-1) is used as the seat material in components that will be exposed to high pressure nitrous oxide (gas source manifolds). The data in this report indicate that either PCTFE, polyimide, or PTFE will perform satisfactorily at pressures of 150 psig or less in nitrous oxide service (VMBs and process tools). Therefore, for low pressure pneumatic valves (such as the AP3540 and AP4540) and mini pressure regulators (such as the AP500) used at less than 150 psig, it is recommended that PTFE or PCTFE are used instead of polyimide (Vespel SP-1) for the seat material because it will reduce cost and increase the long term seat leak integrity of the device.

It should be noted that the data contained in this report cannot duplicate the exposure of seat materials to nitrous oxide in actual use conditions and that a longer duration of exposure may affect the results.

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