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Oxygen Compatibility of Polymers Including TFE-Teflon<sup>®</sup>, Kel-F<sup>®</sup> 81, Vespel<sup>®</sup> SP-21, Viton<sup>®</sup> A, Viton<sup>®</sup> A-500, Fluorel<sup>®</sup>, Neoprene<sup>®</sup>, EPDM, Buna-N, and Nylon 6,6

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**ABSTRACT:** Ten polymeric materials including EPDM, Nylon 6,6, Buna-N and other materials marketed\* as TFE-Teflon<sup>®</sup> (PTFE), Kel-F<sup>®</sup> 81 (PCTFE), Vespel<sup>®</sup> SP-21, Viton<sup>®</sup> A, Viton<sup>®</sup> A-500, Fluorel<sup>®</sup>, and Neoprene<sup>®</sup> were systematically evaluated for their oxygen compatibility property. The specific properties examined included: (1) autoignition temperature (AIT), (2) heat of combustion, and (3) liquid-oxygen (LOX) mechanical impact sensitivity. Test methods and procedures employed for the evaluations were in compliance with those prescribed in the ASTM and BSI standards. Additionally, a BOC in-house high-pressure autoignition test rig (HP-AIT) was used for a detailed study on the materials’ autoignition behavior. This high-pressure vessel is capable of probing the AIT of a material up to 450°C and at oxygen pressure (prior to a thermal excursion) up to 5000 psig (~34.5 MPa).

According to the test results, fluorinated polymers including TFE<sup>®</sup>, Kel-F<sup>®</sup> 81, Viton<sup>®</sup> A, Viton<sup>®</sup> A-500 and Fluorel<sup>®</sup> exhibited superior oxygen compatibility property. Specifically, they possessed relatively high autoignition temperatures and reasonably low heat of combustion values. Among them, TFE<sup>®</sup> possessed the best property by showing the highest autoignition temperature (>450°C), lowest heat of combustion (1517 cal/g), and qualified (pass) LOX impact sensitivity. Vespel<sup>®</sup> SP-21 (15% graphite-filled polyimide) exhibited an autoignition temperature higher than fluorinated elastomers like Fluorel<sup>®</sup> and Viton<sup>®</sup> A, but lower than Kel-F<sup>®</sup> 81 as well as TFE<sup>®</sup>. The variation of oxygen pressure, ranging from 1800 psig (~12.4 MPa) to 5000 psig (~34.5 MPa), was found to exert little influence on the autoignition temperature of the polymers.

The autoignition behavior of Kel-F<sup>®</sup>81, Viton<sup>®</sup> A, Vespel<sup>®</sup> SP-21, and Nylon 6,6 was further investigated, using HP-AIT, as a function of sample quantity and surface area at different oxygen pressure. On the one hand, variation of sample quantity exerted a more pronounced effect on the AITs of Viton<sup>®</sup> A, Nylon 6,6 and Kel-F<sup>®</sup> 81 at various pressures than on Vespel<sup>®</sup> SP-21. A higher autoignition temperature was generally observed from samples with smaller quantities. On the other hand, changes in sample surface area had little influence on the autoignition temperature of the materials in 1800 psig (~12.4 MPa) oxygen.

**KEYWORDS:** Autoignition, Gaseous Oxygen, Liquid Oxygen, Heat of Combustion, Mechanical Impact, Polymers, TFE-Teflon<sup>®</sup> (PTFE), Kel-F<sup>®</sup> 81 (PCTFE), Vespel<sup>®</sup> SP-21, Viton<sup>®</sup> A, Viton<sup>®</sup> A-500, Fluorel<sup>®</sup>, Neoprene<sup>®</sup>, EPDM, Nylon 6,6, Buna-N

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\* refer to appendix-I for information on material’s manufacturer

Oxygen compatibility of polymeric materials is a subject area of great concern to various industries, including chemicals and industrial gases, petrochemicals, metals, machinery manufacturing/fabrication, aerospace, medical, and waste management/environmental, as well as government agencies like NASA, Navy and Air Force. To perform effective assessment on the compatibility of engineering materials with oxygen, experimental evaluations in the form of laboratory, prototype, and/or live-component tests are paramount. However, due to excessive cost and time involved in producing oxygen compatibility data for the diverse engineering issues associated with oxygen manufacturing, distribution and applications, an effective approach is to utilize and cross-reference various oxygen compatibility resources available in the public domain. These resources include technical reports and/or standards issued by such organizations as ASTM (American Society for Testing and Materials), NASA (National Aeronautics and Space Administration), CEN (European Committee for Standardization), and BAM (German Federal Institute for Materials Research and Testing). The major drawback of this approach, however, has been the large variation of data from various resources which employed different apparatuses of varying system configurations and operating procedures. Additionally, the statistically uncertain nature of oxygen compatibility data further complicates the perplexity of a multi-disciplinary engineering problem, which requires critical assessment of oxygen compatibility.

In view of this limitation and the ever growing demands for the oxygen compatibility of polymeric materials for various oxygen applications, it is imperative to develop a consistent and reliable database based on datum generated by a cluster of accredited instruments with the same performance baseline. The development of such information is anticipated to advance our knowledge in selecting polymeric materials for oxygen applications in general, and to pave the way for the development of seal materials for high-pressure, compressed oxygen applications such as cylinder filling.

Polymeric materials are widely used as seal materials in compressed gases/oxygen applications. One of the primary concerns for polymeric sealants in a compressed oxygen environment stems from their autogenous ignition behavior at elevated temperatures. Elevated temperatures are attainable by a few mechanisms, with adiabatic compression heating being the most plausible one, which may be introduced by inadvertent operating procedures. Most polymers exhibit relatively lower autoignition temperatures, compared to their metallic counterparts, which in turn renders their surrounding materials susceptible to a promoted combustion event. In general, most combustible polymers have autoignition temperatures in the range of 150 to 500°C in pure oxygen, as compared to 900 to 2000°C for metals [1]. Additionally, their heat of combustion and mechanical impact sensitivity to an LOX (liquid oxygen) environment further complement the autoignition property and play important roles in the critical assessment of oxygen compatibility of polymers.

Up to this time, fair amounts of studies have been dedicated to understand the oxygen compatibility properties of polymers [2-13]. On the one hand, for example, extensive material testing database have been established at NASA on the compatibility of materials with liquid oxygen under the influence of mechanical impact [2-4]. On the other hand, in the case of autoignition property, little understanding was established on

the autoignition behavior of polymers in high pressure oxygen, greater than 2000 psig (~13.8 MPa). According to the study by Nihart and Smith [5], the autoignition (or spontaneous ignition) temperatures of fluorocarbon plastics and elastomers were similar in 7500 psi (~51.7 MPa) and in 2000 psi (~13.8 MPa) oxygen. Furthermore, McGuire's study [14] on a number of polymeric materials, including plastic and elastomeric materials, revealed that their spontaneous ignition temperatures generally decreased with increasing oxygen pressure, ranging from 5 to 40 MPa.

It is not the intent of this work to exhaust literature search on this subject or to compare/correlate data generated by different institutes based on various vessel designs and operating procedures. Instead, the objective is to characterize the oxygen compatibility of polymers based on a systematic study using BOC in-house facility. To satisfy the general needs of oxygen applications, ten polymeric materials were selected and evaluated, with primary emphasis placed on their autoignition behavior. In addition, heat of combustion and LOX mechanical impact sensitivity were evaluated. The materials selected for evaluation are those commonly employed for oxygen service, which included TFE-Teflon<sup>®</sup>, Kel-F<sup>®</sup> 81 (PCTFE), Vespel<sup>®</sup> SP-21, Viton<sup>®</sup> A, Viton<sup>®</sup> A-500, Fluorel<sup>®</sup>, Neoprene<sup>®</sup>, EPDM, Nylon 6,6, and Buna-N. The generic chemical names of and manufacturers for the polymeric materials are listed in Appendix-I for reference.

#### \*APPENDIX-I Materials Evaluated in this Study

MATERIAL	GENERIC CHEMICAL NAME	MANUFACTURER/ SUPPLIER
TFE-Teflon <sup>®</sup>	PTFE/polytetrafluoroethylene	DuPont
Kel-F <sup>®</sup> 81	PCTFE/polychlorotrifluoroethylene	3 M
Vespel <sup>®</sup> SP-21	15% graphite filled polyimide	DuPont
Viton <sup>®</sup> A/Viton <sup>®</sup> A500	vinylidene fluoride hexafluoropropylene	DuPont
Fluorel <sup>®</sup>	vinylidene fluoride hexafluoropropylene	3 M
Neoprene <sup>®</sup>	polychloroprene	DuPont
EPDM	ethylene propylene-diene terpolymers	DuPont
Nylon 6,6	polyamide	DuPont
Buna-N	nitrile butadiene	N.A./McMaster-Carr

## EXPERIMENTAL PROCEDURES

The specific properties measured from these polymeric materials included (1) autoignition/spontaneous-ignition temperature (AIT/SIT) at oxygen pressures ranging from 1800 psig (~12.4 MPa) to 5000 psig (~34.5 MPa), (2) LOX impact sensitivity at atmospheric pressure under mechanical impact energy 122 Joules, and (3) heat of combustion at oxygen pressure 25 atm (~2.5 MPa or 367.5 psig ).

In the study of autoignition behavior, most efforts were spent on the BOC/GTC newly-installed high-pressure autoignition testing equipment (HP-AIT ) because of its high pressure bearing capability- up to 12,000 psig (~82.8 MPa) at 460°C. The characteristics and functionality of this HP-AIT are briefly described in Appendix-II. According to the system's capability, four different pressure settings  $P(i)=1800$  psig (~12.4 MPa), 3000 psig (~20.7 MPa), 4000 psig (~27.6 MPa) and 5000 psig (~34.5 MPa) were selected for the determination of autoignition temperature of each polymer, where  $P(i)$  denotes the initial oxygen pressure in the test vessel at ambient condition prior to temperature excursion. The final pressure  $P(f)$  of the vessel at the onset of an autoignition behavior cannot be regulated based on its current design, and is chiefly determined by the volume and final temperature of the vessel. To gain statistical confidence on the autoignition temperature data, three duplicate tests were performed on each material at every testing condition.

As baseline information, autoignition behavior of the polymeric materials was also investigated using an ASTM [15] and a BSI (British Standards Institution) [9,16] standardized testing vessels at two different pressure settings,  $P(i)=1500$  psig (~10.3 MPa) and 1800 psig (~12.4 MPa), respectively. The sample heating rates required by the ASTM and BSI standard tests were 5 and 20°C respectively, as compared to 20°C selected for the HP-AIT tests. In addition, three and six duplicate tests, respectively, were performed on each material at every testing condition for the ASTM and BSI standard tests. In a manner similar to HP-AIT, the final oxygen pressures in both the ASTM and BSI pressure vessels were not controllable. Detailed operating procedures for the ASTM and BSI autoignition tests were in full compliance with the guidelines prescribed in the ASTM G-72 [15] and BS 3N 100 [16] standards, respectively.

The amounts of sample quantity employed for the ASTM and BSI tests were about 200 and 60 mg, respectively, as regulated by the respective standards. Based on a prior calibration study by Swindells et al. [9] using a BS 3N 100 pressure vessel, samples were sliced into at least 6 pieces of a similar size in order to yield consistent and conservative measurements. In the case of BOC in-house HP-AIT, since it is not a standardized test vessel under any standard institutions, samples of 60 mg quantity were selected for general-purpose autoignition studies.

Additional tests were performed on selected materials of various quantities, ranging from 5 to 120 mg, with an objective to understand the effect of sample quantity on the autoignition temperature of the materials at different oxygen pressure. In these tests, samples were typically sliced into at least six pieces in order to be consistent with the

practices adopted for the ASTM and BSI tests. Furthermore, the effect of sample surface area on AIT was investigated on specific materials by dissecting samples of 60 mg in weight into 2, 6, 12, 20 and 30 test pieces of a uniform size. Through these studies, we anticipate not only to produce results for high-pressure oxygen applications, but also to fully comprehend the performance of the HP-AIT system for future work in material selection and development.

As for the LOX mechanical impact tests and heat of combustion measurements, they were carried out in full compliance with the guidelines prescribed in the BS 3N 100 [10,16] and ASTM D3286 [17] standards, respectively. The operating procedures for these standardized testing are cumbersome but straightforward to follow, which will not be further described in this report. Nevertheless, it is important to point out the following. According to the BS 3N 100, the LOX impact test is a pass or fail trial. In order to pass the test, a material shall exhibit no audible detonation or not more than one visible ignition in 10 tests on fresh samples. As for the heat of combustion measurements, they were performed by a commercial laboratory- Mineral Labs., Inc: in Salyersville, Kentucky. Three duplicate tests were conducted on each material, and their arithmetic mean value calculated as the characteristic heat of combustion of the material.

## APPENDIX-II: BOC High-Pressure Autoignition Test Apparatus

### Pressure Vessel Specifications:

Body/Cover/Main Nut Materials:	Monel K500
Dimension (in)/Capacity (cc):	7/8" I.D. x 3 7/8" I.L./38 cc
Max. Working Pressure:	82.75 MPa (12,000 psig)
Max. Working Temperature:	460°C
Orientation:	Vertical
Hydrotest Pressure:	124.13 MPa (18,000 psig) for 10 mins

### Air-Operated Compressor Specifications:

Max. Discharge Pressure:	69.96 MPa (10,000 psig)
(Air Pressure Required:	90 psig)
Body Material:	Monel 400
Piston Material:	Monel K500
Piston Diameter:	7/16"
Stroke Length:	4"

### Testing Parameters:

Pressure:	~34.5 MPa (5000 psig*)
Heating Rate:	Variable (20°C/min used for this study)
Sample Environment:	Gaseous oxygen with thermal excursion
Gas Flow:	Static or Constant pressure**
No. of Test Required:	3 consecutive tests
Max. Test Temp.:	450°C

\* prior to thermal excursion; \*\* through the control of a back pressure regulator

## RESULTS AND DISCUSSION

### I. AUTOIGNITION BEHAVIOR

#### (A) Autoignition Temperature vs. Oxygen Pressure

Detailed experimental results on the AITs/SITs of polymeric materials, as a function of oxygen pressure, are presented in Table I. Also shown in Table I are initial pressures before temperature excursion, P(i), and final pressures at the onset of autoignition, P(f), of the pressure vessel chambers for each material of test. According to the test results determined by HP-AIT, the performance of the polymeric materials can be rated in a descending order as TFE-Teflon® > Kel-F® 81 > Vespel® SP-21 > Fluorel® > Viton® A/Viton® A-500 > EPDM > Nylon 6,6 > Neoprene®/Buna-N. The rating is valid for oxygen pressures at 1800 psig (~12.4 MPa), 3000 psig (~20.7 MPa), 4000 psig (~27.6 MPa), and 5000 psig (~34.5 MPa). Viton A and Viton A-500 as well as Neoprene and Buna-N, respectively, appeared to exhibit similar AITs at all the oxygen pressures examined.

As for the AITs measured by the ASTM and BSI pressure vessels at 1500 psig (~10.3 MPa) and 1800 psig (~12.4 MPa) respectively, slight inconsistency in the materials' rating was noted. On the one hand, according to the BSI test results, the materials can be ranked as TFE-Teflon® > Kel-F® 81 > Vespel® SP-21 > Viton® A-500/Fluorel® > Viton® A > Neoprene® > EPDM > Nylon 6,6 > Buna-N. On the other hand, as inferred by the ASTM results, the materials can be rated as TFE-Teflon® > Kel-F® 81 > Vespel® SP-21 > Fluorel® > Neoprene® > Viton® A/A-500 > EPDM > Nylon 6,6 > Buna-N. **Above all, TFE-Teflon®, Kel-F® 81 and Vespel® SP-21 are consistently rated as the most oxygen compatible materials among all; EPDM, Nylon 6,6 and Buna-N are consistently rated as the least compatible materials.**

By comparing the data generated by ASTM and BSI vessels at their designated test pressures, as well as by HP-AIT at 1800 psig (~12.4 MPa), ASTM vessel appeared to have produced the most conservative autoignition temperature, followed by BOC's HP-AIT vessel and then BSI vessel. An exceptional case, however, was noted for Neoprene® whose AIT was the lowest according to HP-AIT tests, followed by the ASTM and BSI tests.

For the ease of rating materials in terms of their autoignition temperatures (as measured by HP-AIT) and to better illustrate the dependence of their AITs on oxygen pressure, data shown in Table I are graphically displayed in Fig. 1. It should be noted that Viton® A-500 is not presented in Fig. 1 for clarity reason, because of its similarity in AIT to Viton® A. As revealed by Fig. 1, the AITs of various polymeric materials appeared to be rather insensitive to pressure variation, although a slight influence seemed to have been noted in Vespel® SP-21, Fluorel®, and Viton® A. In consideration of the small sampling size (three duplicate tests) used for the calculation of an effective AIT, it is difficult to clarify whether the variation of AIT, as a function of oxygen pressure, is statistically significant enough to be considered as an intrinsic material property.

Table I. Autoignition temperatures of polymeric materials measured by ASTM G72, BS 3N 100 and BOC high-pressure testing vessels

Property Material	ASTM-AIT (°C) (200 mg/ 3 tests)	BSI- SIT (°C) (60 mg/ 6 tests)	BOC High Pressure-AIT (°C) (60 mg/30 mg/3 tests)			
	P(i), psig P(f), psig	1500 1800-2200	1800 2000-2400	1800 2600-3200	3000 4300-5500	4000 5900-7500
TFE* (PTFE)	425+/425+/ 425+  AIT = 425+ P(f)=2200	400+/400+ 400+/400+ 400+/400+  SIT = 400+ P(f)=2400	450+ 450+ 450+ (**) AIT = 450+  P(f)=3000	450+ 450+ 450+ (**) AIT = 450+  P(f)=5200	450+ 450+ 450+ (**) AIT = 450+  P(f)=7500	450+ 450+ 450+ (**) AIT = 450+  P(f)=10200
Kel-F* 81 (PCTFE)	404.3/404.3/ 404.3  AIT = 404 P(f)=2200	400+/400+ 400+/400+ 400+/400+  SIT = 400+ P(f)=2300	417/414/ 417  AIT = 412 P(f)=3000	418/418/ 418  AIT = 418 P(f)=5200	420/419/ 425  AIT = 414 P(f)=7200	430/426/428 (432/435/439)++  AIT = 424 (428)++ P(f)=9500 (9900)++
VespeI* SP-21	347.2/347.2/ 347.2  AIT = 347 P(f)=2100	400+/400+ 400+/400+ 400+/400+  SIT = 400+ P(f)=2400	383/383/ 385  AIT = 361 P(f)=3200	369/364/ 367  AIT = 361 P(f)=5500	357/356/ 358  AIT = 355 P(f)=7100	352/355/355 (350/349/348)++  AIT = 350 (347)++ P(f)=8800 (9000)++
Viton* A	252.0/254.4/ 244.1  AIT = 239 P(f)=2000	394/400+(* 389/403 375/381  SIT = 368 P(f)=2300	255/256/ 260  AIT = 251 P(f)=2700	267/264/ 260  AIT = 256 P(f)=4700	259/263/ 266  AIT =255 P(f)=6700	268/272/274 (328/317/328)++  AIT =265 (311)++ P(f)=8600 (9200)++
Viton* A-500	248.0/243.7/ 243.0  AIT = 239 P(f)=2000	391/400+(* 396/390 394/400+(*  SIT = 386 P(f)=2400	258/263/ 262  AIT = 255 P(f)=2900	264/259/ 256  AIT = 251 P(f)=5100	259/257/ 266  AIT = 251 P(f)=6500	254/254/263 (331/332/333)++  AIT = 246 (330)++ P(f)= 8600 (9100)++

P(i) denotes initial pressure before thermal excursion;

P(f) denotes final pressure at the onset of autoignition;

+ Autoignition temperature greater than 400°C; \* 400°C was used for AIT calculation;

@ sample melted without ignition; # sample evaporated without ignition;

\*\* particulate samples fused together;

++ Data in parentheses were measured in 5000 psig O<sub>2</sub> using 30 mg sample.

Table I. Autoignition temperatures of polymeric materials measured by ASTM G72, BS 3N 100 and BOC high-pressure testing vessels (continued)

Property Material	ASTM-AIT (°C) (200 mg/ 3 tests)	BSI- SIT (°C) (60 mg/ 6 tests)	BOC High Pressure-AIT (°C) (60 mg/30 mg/3 tests)			
			1800 2600-3200	3000 4300-5500	4000 6000-7500	5000 (***) 7300-10200
<b>Fluorel®</b>	307.6/321.8/ 309.6  AIT = 297 P(f)=2100	388/396 400/393 387/398  SIT = 383 P(f)=2400	338/338/ 335  AIT = 333 P(f)=3100	337/334/ 334  AIT = 331 P(f)=5300	330/332/ 335  AIT = 327 P(f)=7000	321/326/330 (331/331/329) <sup>++</sup>  AIT = 316 (328) <sup>++</sup> P(f)=8800 (8900) <sup>++</sup>
<b>Neoprene®</b>	262.7/255.9/ 249.5  AIT = 242 P(f)=2000	319/319 314/318 325/310  SIT = 307 P(f)=2300	184/192/ 184  AIT = 177 <sup>a</sup> P(f)=2600	185/186/ 184  AIT = 183 <sup>b</sup> P(f)=4400	184/185/ 186  AIT = 183 <sup>b</sup> P(f)=5900	180/180/179 (182/184/184) <sup>++</sup>  AIT = 178 (181) <sup>b++</sup> P(f)=7400 (7500) <sup>++</sup>
<b>EPDM</b>	215.8/209.5/ 214.4  AIT = 206 P(f)=2100	278/277 275/277 259/283  SIT = 258 P(f)=2300	219/219/ 221  AIT = 217 P(f)=2800	219/221/ 221  AIT = 218 P(f)=4700	219/215/ 219  AIT = 213 P(f)=6200	216/219/214 (214/215/216) <sup>++</sup>  AIT = 211 (213) <sup>++</sup> P(f)=7800 (8000) <sup>++</sup>
<b>Nylon 6,6</b>	182.1/189.5/ 190.9  AIT = 178 P(f)=2000	247/245 244/243 239/239  SIT = 236 P(f)=2000	205/203/ 208  AIT = 200 P(f)=2800	201/200/ 201  AIT = 199 P(f)=4500	192/197/ 198  AIT = 189 P(f)=6000	193/194/195 (198/194/197) <sup>++</sup>  AIT = 192 (192) <sup>++</sup> P(f)=7500 (7600) <sup>++</sup>
<b>Buna-N</b>	173.8/169.9/ 169.9  AIT = 166 P(f)=1800	225/228 225/230 233/232  SIT = 222 P(f)=2200	191/191/ 191  AIT = 191 P(f)=2800	192/189/ 184  AIT = 180 P(f)=4300	183/181/ 184  AIT = 179 P(f)=6000	184/182/183 (184/182/185) <sup>++</sup>  AIT = 181 (180) <sup>++</sup> P(f)=7400 (7300) <sup>++</sup>

P(i) denotes initial pressure before thermal excursion;

P(f) denotes final pressure at the onset of autoignition

**a**: blackish residues observed; **b**: no blackish residues observed**++** Data in parentheses were measured in 5000 psig O<sub>2</sub> using 30 mg sample.



# Effect of Oxygen Pressure on the AIT of Polymers

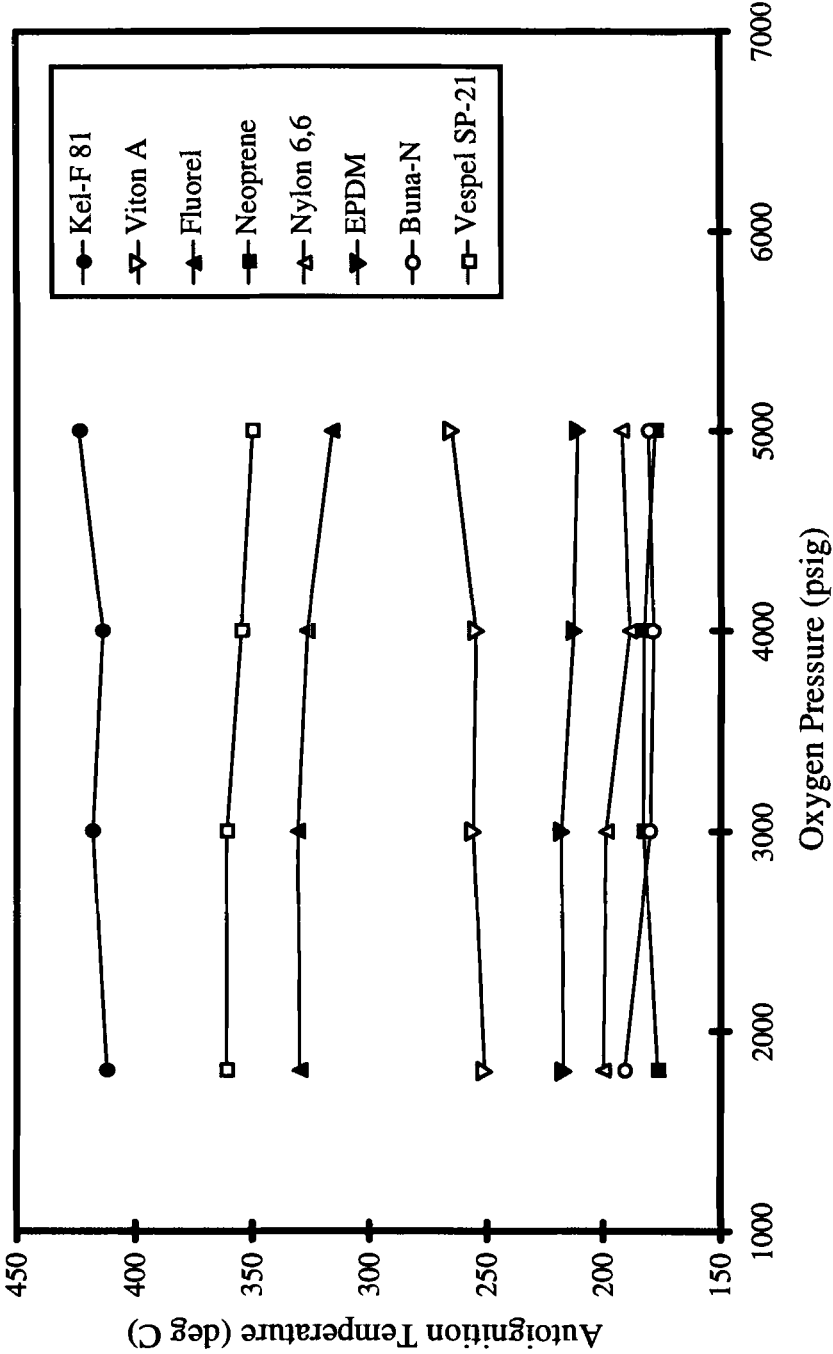


Fig.1 The effect of oxygen pressure on the autoignition temperature of polymers

The present experimental results are consistent with the data shown in an earlier study by Nihart and Smith [5] using the pressure vessel setup at Union Carbide. According to the study, the spontaneous ignition temperatures of fluorocarbon plastics and elastomers (Viton A & B, Teflon, Rulon A, B & C, Duroid 5600, 5650, 5870, 5813, Kel-F81, Kel-F elastomer 3700 & 5500, and Neoprene) were similar in 7500 psi (~51.7 MPa) and in 2000 psi (~13.8 MPa) oxygen. On the other hand, McGuire's study [14] at the South Bank University, based on a high-pressure vessel with rather different design configurations from the BOC's vessel, revealed that the autoignition temperatures of some polymeric materials decreased with increasing oxygen pressure, ranging from 5 to 40 MPa. The materials studied in reference 14 included polyurethane, Nylon 6,6, polybutadiene, Vespel SP-21, Fluorel, Aflas, Viton, PTFE, Neoprene, PCTFE, chloroprene and silicone rubber. According to McGuire's study, the extent of pressure-effect on the variation of AIT is material dependent, and it can be qualitatively described by an equation:  $1/T_{AIT} \sim \ln P$ . Since ignition and combustion of materials in oxygen involve intricate reactions and are greatly influenced by even moderate changes in the environmental and geometrical conditions, it is difficult to provide a reasoning base for the differential autoignition behavior of these polymeric materials under different test configurations.

### (B) Autoignition Temperature vs. Sample Quantity and Oxygen Pressure

Based on the rating of the materials and their popularity being specified for oxygen applications, Kel-F® 81, Vespel® SP-21, Fluorel®, Viton® A, EPDM and Nylon 6,6 were further evaluated using HP-AIT for autoignition behavior in 1800 psig (~12.4 MPa) oxygen, as a function of sample quantity. The objectives of conducting this study using HP-AIT are twofold: (1) to comprehend the effects of the amount of polymeric material on autoignition temperature; and (2) to better understand and gauge the performance and functionality of this new pressure vessel in order to pave the way for future studies at higher oxygen pressures.

Detailed experimental results on the AITs of these materials are presented in Table II and graphically displayed in Fig.2. As revealed in Fig.2, there exists a clear and consistent trend for the AITs of Kel-F® 81, Vespel® SP-21, Viton® A and Nylon 6,6 to decrease with increasing sample quantity. This phenomenon was noted to be more pronounced for Viton® A, Nylon 6,6 and Kel-F® 81, than for Vespel® SP-21. In most cases, the transition of AITs appeared to have taken place in regions where the amounts of sample quantity were less than ~30 mg. By increasing the sample quantities to 30 mg or greater, the AITs of most materials tended to stabilize and stayed as constant values.

In consideration of the widespread applications of Viton® A, Kel-F® 81, and Nylon 6,6, as well as the potential of Vespel® SP-21, as polymeric seal materials in various oxygen applications, it is essential to further explore such an intriguing behavior of these materials at higher oxygen pressures. As a result, parametric studies were carried out to investigate the effects of sample quantity and oxygen pressure on the autoignition behavior of Viton® A, Kel-F® 81, Nylon 6,6, and Vespel® SP-21. Experimentally, the autoignition temperature of these materials was evaluated as a function of sample weights

(5, 10, 20, 30, 60 and 120 mg) at various oxygen pressures including 1800 psig (~12.4 MPa), 3000 psig (~20.7 MPa), 4000 psig (~27.6 MPa), and 5000 psig (~34.5 MPa). The experimental results obtained from Viton® A, Kel-F® 81, Nylon 6,6, and Vespel® SP-21 are presented in Tables III, IV, V, and VI respectively, and graphically displayed in Figs. 3, 4, 5, and 6 respectively.

Table II. Effect of sample quantity on the autoignition temperature of some polymers in 1800 psig oxygen

**Effect of Sample Quantity on the AIT of Viton® A**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	350	353	355	352.6667	2.516611	5.033223	347.6334
10	349	345	351	348.3333	3.05505	6.110101	342.2232
20	270	268	269	269	1	2	267
30	263	264	268	265	2.645751	5.291503	259.7085
60	258	263	262	261	2.645751	5.291503	255.7085
120	259	255	254	256	2.645751	5.291503	250.7085

**Effect of Sample Quantity on the AIT of EPDM**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	238	234	234	235.3333	2.309401	4.618802	230.7145
10	234	229	229	230.6667	2.886751	5.773503	224.8932
20	222	224	223	223	1	2	221
30	220	222	220	220.6667	1.154701	2.309401	218.3573
60	219	219	221	219.6667	1.154701	2.309401	217.3573
120	219	220	222	220.3333	1.527525	3.05505	217.2783

**Effect of Sample Quantity on the AIT of Nylon 6,6**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	209	211		210	1.414214	2.828427	207.1716
10	207	205	203	205	2	4	201
20	202	201	202	201.6667	0.57735	1.154701	200.512
30	200	202	199	200.3333	1.527525	3.05505	197.2783
60	205	203	208	205.3333	2.516611	5.033223	200.3001
120	190	192	193	191.6667	1.527525	3.05505	188.6116
5	249	249	254	250.6667	2.886751	5.773503	244.8932
10	249	249	252	250	1.732051	3.464102	246.5359

**Note:** all sample weights in mg; all temperatures in °C;  
triplicate measurements were performed on each material

Table II. Effect of sample quantity on the autoignition temperature of some polymers in 1800 psig oxygen (continued)

**Effect of Sample Quantity on the AIT of Kel-F® 81**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	448	449	449	448.6667	0.57735	1.154701	447.512
10	434	431	442	435.6667	5.686241	11.37248	424.2942
20	423	422	423	422.6667	0.57735	1.154701	421.512
30	421	417	419	419	2	4	415
60	417	414	417	416	1.732051	3.464102	412.5359
120	418	418	413	416.3333	2.886751	5.773503	410.5598

**Effect of Sample Quantity on the AIT of Fluorel®**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	338	335	333	335.3333	2.516611	5.033223	330.3001
10	335	331	329	331.6667	3.05505	6.110101	325.5566
20	332	329	330	330.3333	1.527525	3.05505	327.2783
30	329	330	333	330.6667	2.081666	4.163332	326.5033
60	338	338	335	337	1.732051	3.464102	333.5359
120	325	330	328	327.6667	2.516611	5.033223	322.6334

**Effect of Sample Quantity on the AIT of Vespel® SP-21**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	386	385	390	387	2.645751	5.291503	381.7085
10	385	384	380	383	2.645751	5.291503	377.7085
20	373	369	369	370.3333	2.309401	4.618802	365.7145
30	370	367	368	368.3333	1.527525	3.05505	365.2783
60	363	364	362	363	1	2	361
120	358	359	366	361	4.358899	8.717798	352.2822

**Note:** all sample weights in mg; all temperatures in °C;  
triplicate measurements were performed on each material

# Effect of Sample Quantity on the AIT of Polymers in 1800 psig Oxygen

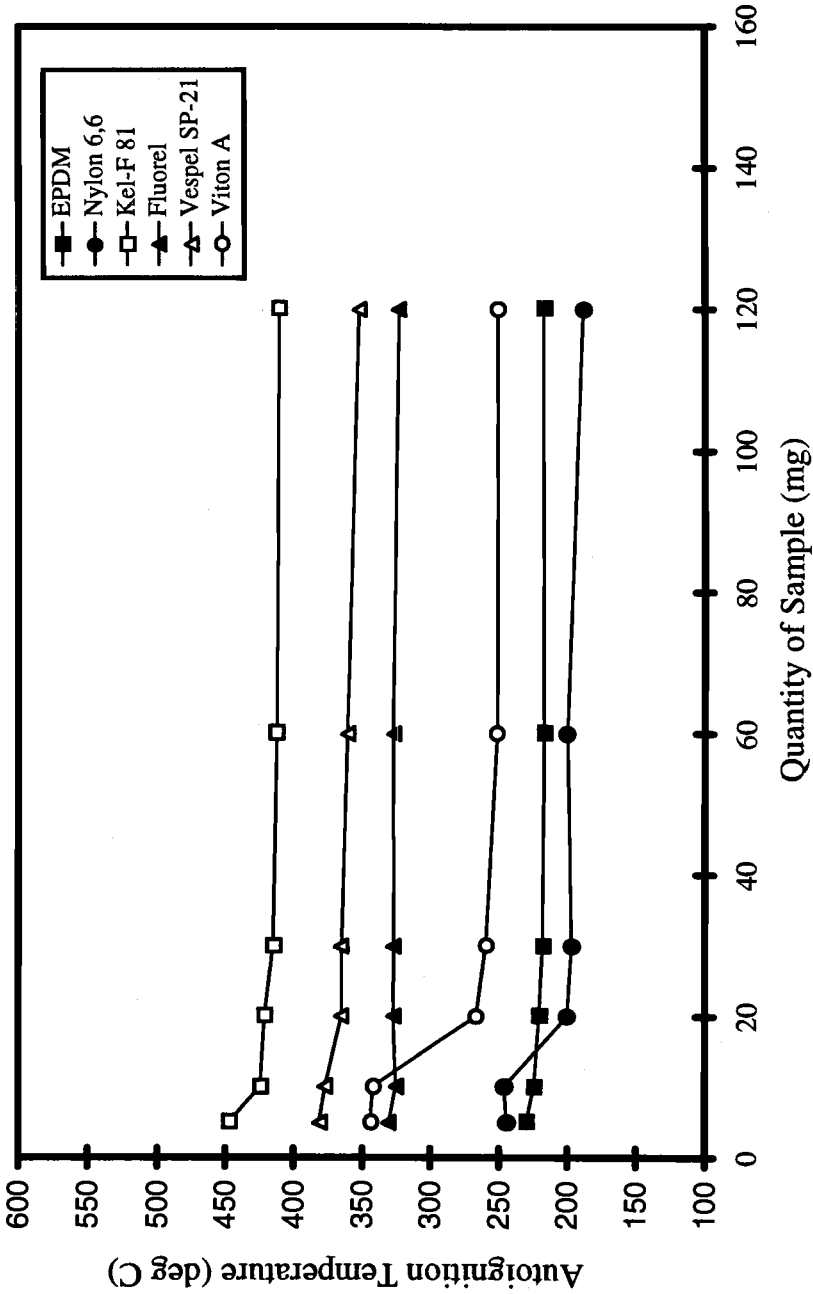


Fig. 2 The effect of sample quantity on the autoignition temperature of polymers in 1800 psig oxygen

### Viton® A

As shown in Table III and Fig.3, Viton® A clearly exhibited a strong dependence of AIT on the sample quantities of test. For all the pressures evaluated, the AITs of Viton® A scattered in two distinctly different regions with a transition zone falling on sample quantities between 10 and 35 mg. As a general characteristic behavior, higher autoignition temperatures ranging from 330 to 350°C were noted from samples with quantities smaller than 20 mg; lower autoignition temperatures between 250 and 260°C were observed from samples with quantities greater than 35 mg. As delineated by the curves shown in Fig.3, the AITs of Viton A tended to shift toward a higher temperature regime with increasing oxygen pressure for the same amount of sample quantity. The material eventually exhibited a fairly constant AIT value when the sample quantities reached 60 and 120 mg.

A similar autoignition behavior was also observed from Viton® A-500 in 5000 psig (~34.5 MPa) oxygen. As shown in Table I, the AITs of Viton® A-500 also transited from 330 to 246°C when the sample quantities varied from 30 and 60 mg, respectively. Although a parametric study on the combined effect of sample quantity and oxygen pressure was not performed on Viton A-500, the transition behavior of AIT is expected to occur in this material as well, because of the similarity in chemistry between Viton A and Viton® A-500. As an "A-type" Viton®, both Viton® A and Viton® A-500 are dipolymers of hexafluoropropylene (HFP) and vinylidene fluoride (VF2), and contain 66% fluorine. The primary differences between them lie in their nominal viscosity and compression set; Viton® A and Viton® A-500 exhibit viscosity (ML 1+10 at 121°C) 60 and 50 respectively, and compression set (70 hrs at 200°C) 25% and 15% respectively.

The presently observed dual-AIT property of Viton® A and Viton® A-500 is very intriguing and has not been reported in any published literature. This unique behavior is clearly strongly dependent upon sample quantity and, to a lesser extent, on oxygen pressure. Such a material characteristic may be attributable to the material chemistry of Viton® A, whose chemical constituents HFP and VF2 are expected to exhibit two distinctly different autoignition temperatures. According to literature data, polyvinylidene fluorides exhibit AITs ranging from 243 to 311°C, while no information is available on HFP. It is likely that the dipolymer nature of the "A-type" Viton® played a role in displaying two different autoignition temperatures when the sample quantity is small.

Table III. Effect of sample quantity on the autoignition temperature of Viton® A at various oxygen pressures

**Effect of Sample Quantity on the AIT of Viton® A in 1800 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	350	346	350	348.6667	2.309401	4.618802	344.0479
10	349	345	351	348.3333	3.05505	6.110101	342.2232
20	270	268	269	269	1	2	267
30	263	264	268	265	2.645751	5.291503	259.7085
60	255	256	260	257	2.645751	5.291503	251.7085
120	259	255	254	256	2.645751	5.291503	250.7085

**Effect of Sample Quantity on the AIT of Viton® A in 3000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	352	349	360	353.6667	5.686241	11.37248	342.2942
10	348	343	342	344.3333	3.21455	6.429101	337.9042
20	340	336	335	337	2.645751	5.291503	331.7085
30	271	272	275	272.6667	2.081666	4.163332	268.5033
60	267	264	260	263.6667	3.511885	7.023769	256.6429
120	257	255	256	256	1	2	254
30	339	0	0	339	0	0	339

**Effect of Sample Quantity on the AIT of Viton® A in 4000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	344	347	344	345	1.732051	3.464102	341.5359
10	339	340	340	339.6667	0.57735	1.154701	338.512
20	332	338	338	336	3.464102	6.928203	329.0718
30	269	267	268	268	1	2	266
60	259	263	266	262.6667	3.511885	7.023769	255.6429
120	259	258	258	258.3333	0.57735	1.154701	257.1786

**Effect of Sample Quantity on the AIT of Viton® A in 5000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	357	352	354	354.3333	2.516611	5.033223	349.3001
10	350	349	344	347.6667	3.21455	6.429101	341.2376
20	343	342	340	341.6667	1.527525	3.05505	338.6116
25	338	328	341	335.6667	6.806859	13.61372	322.0529
30	267	270		268.5	2.12132	4.24264	264.2574
60	268	272	274	271.3333	3.05505	6.110101	265.2232
100	264			264	0	0	264
120	260			260	0	0	260
30	328	317	328	324.3333	6.350853	12.70171	311.6316
35	331	331	336	332.6667	2.886751	5.773503	326.8932

**Note: all sample weights in mg; all temperatures in °C;  
triplicate measurements were performed on each material**

# Effect of Oxygen Pressure and Sample Quantity on the AIT of Viton A

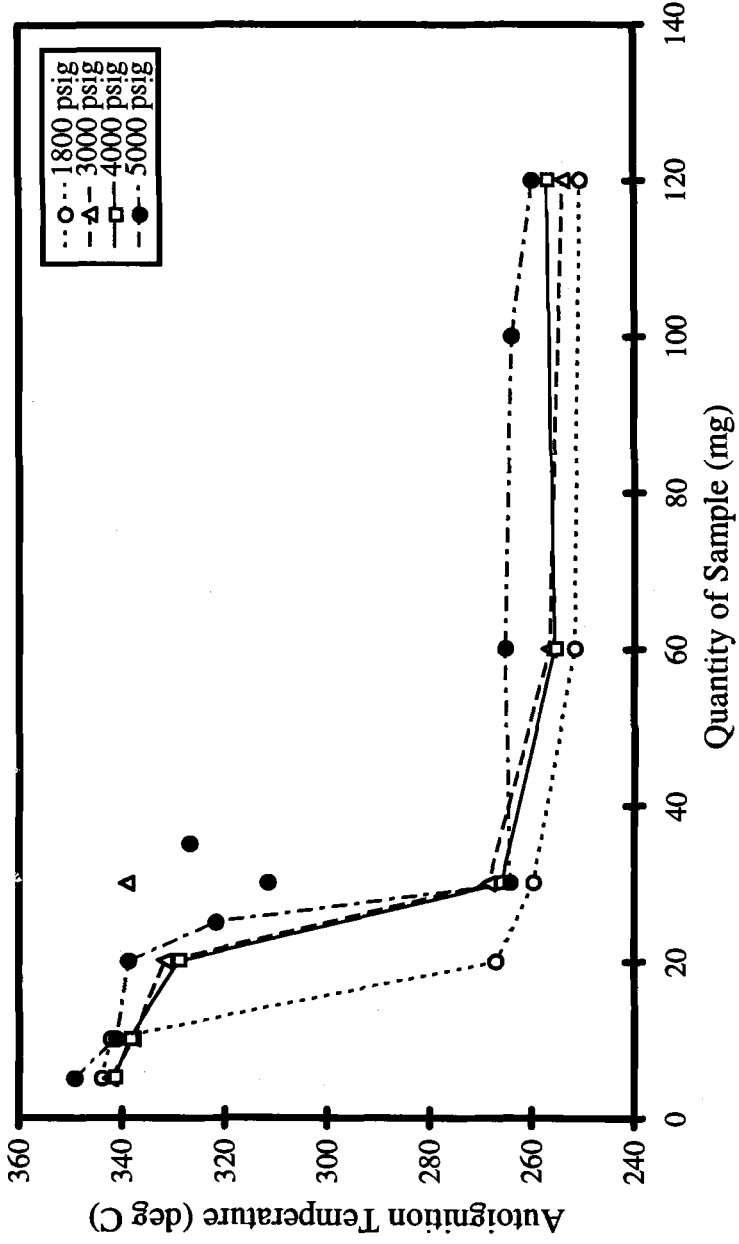


Fig.3 The effect of sample quantity on the autoignition temperature of Viton® A at various oxygen pressures



### **Kel-F® 81**

Experimental results on the effects of sample quantity on the AITs of Kel-F® 81 at various oxygen pressure are presented in Table IV and graphically depicted in Fig.4. At all the oxygen pressures studied, including 1800 psig (~12.4 MPa), 3000 psig (~20.7 MPa), 4000 psig (~27.6 MPa), and 5000 psig (~34.5 MPa), not a single autoignition event was detected from the 5mg samples when the temperature of the pressure vessel was escalated up to 465°C. All the samples appeared to have disappeared, possibly by thermal decomposition, when the sample vials were retrieved for inspection after the tests.

Distinct autoignition temperature was detected from samples when their quantities reached 10 mg or greater. The AITs measured from 10 mg samples were in the range of 440-450°C, which descended further with increasing sample quantity and leveled off at about 420-425°C after sample quantity reaching 30 mg or greater.

The disappearance of an autoignition temperature in Kel-F® 81 at a smaller quantity and reappearance of AITs in larger quantity samples may serve as an indirect proof that it is the **vapor phase** which governs the autoignition behavior of some polymeric materials, such as Kel-F® 81. It is postulated that a threshold vapor concentration is required for a specific material in order to effect an autoignition event of the material. While this hypothesis is yet to be verified, it has long been believed that the combustion of organic materials including polymers and hydrocarbons takes place in a vapor state.

### **Nylon 6,6**

The autoignition behavior of Nylon 6,6 was also examined as a function of sample quantity at four different levels of oxygen pressures. As indicated in Table V and Fig.5, the dependence of AITs on sample quantity appeared to be influenced by oxygen pressure. A much more pronounced effect was noted in 1800 psig (~12.4 MPa) oxygen than in other oxygen pressures investigated. Specifically, an AIT of ~245°C on average was measured in 1800 psig (~12.4 MPa) oxygen from the samples of 5 and 10 mg in quantity. This is in contrast to what was observed from Viton® A and Kel-F® 81, where the effect of sample quantity on AITs appeared to be insensitive to oxygen pressure. The autoignition temperature of Nylon 6,6 reached a constant value, regardless of the oxygen pressure, when the sample quantities are equal to or greater than 20 mg.

### **Vespel® SP-21**

The superior mechanical property (comparable to Nylon 6,6 but significantly better than PTFE and Kel-F®) and decent oxygen compatibility property, as demonstrated by its autoignition temperature, makes Vespel® SP-21 an attractive candidate material for sealing applications in high-pressure oxygen. In view of its potential for applications in oxidizing gases, it is beneficial to explore further the effects of sample quantity and oxygen pressure on the autoignition behavior of Vespel® SP-21.

Table VI summarizes the experimental results on the effects of sample quantity on the AITs of Kel-F® 81 at different oxygen pressure. The results are graphically displayed in Fig.6 for the ease of characterizing the autoignition behavior. According to Fig.6, the AITs of Vespel® SP-21 decreased with increasing sample quantity. The degree of

influence appeared to have been affected by oxygen pressure. Significantly higher AITs, ~375°C, were measured from the 5 mg samples at 1800 psig (~12.4 MPa) and 3000 psig (~20.7 MPa) than those, ~350°C, were measured at 4000 psig (~27.5 MPa) and 5000 psig (~34.5 MPa). Furthermore, the material appeared to exhibit two levels of effective AITs, depending on oxygen pressure, as they reached a constant value at a larger sample quantity. The AITs of Vespel® SP-21 leveled off at ~360°C in 1800 psig (~12.4 MPa) oxygen. The plateau descended to about 335-340°C with increasing oxygen pressure. This behavior is consistent with the property exhibited in Table I and Figure 1.

**Table IV. Effect of sample quantity on the autoignition temperature of Kel-F® 81 at various oxygen pressures**

**Effect of Sample Quantity on the AIT of Kel-F® 81 in 1800 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2&amp;3</u>	<u>Temp4</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5*	>450	>450	>465				>465*
10	452	448	458	452.6667	5.033223	10.06645	442.6002
20	450	443	444	445.6667	3.785939	7.571878	438.0948
30	430	432	440	434	5.291503	10.58301	423.417
60	430	438	434	434	4	8	426
120	422	421	426	423	2.645751	5.291503	417.7085

**Effect of Sample Quantity on the AIT of Kel-F® 81 in 3000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5*	>465	>465	>465				>465*
10	452	452	453	452.3333	0.57735	1.154701	451.1786
20	443	438	449	443.3333	5.507571	11.01514	432.3182
30	447	433	434	438	7.81025	15.6205	422.3795
60	432	430	433	431.6667	1.527525	3.05505	428.6116
120	431	426	430	429	2.645751	5.291503	423.7085

**Effect of Sample Quantity on the AIT of Kel-F® 81 in 4000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5*	>465	>465	>465				>465*
10	461	450	468	459.6667	9.073772	18.14754	441.5191
20	452	450	453	451.6667	1.527525	3.05505	448.6116
30	437	435	444	438.6667	4.725816	9.451631	429.215
60	436	432	432	433.3333	2.309401	4.618802	428.7145
120	423	426	430	426.3333	3.511885	7.023769	419.3096

**Effect of Sample Quantity on the AIT of Kel-F® 81 in 5000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5*	>465	>465	>465				>465*
10	463	465	456	461.3333	4.725816	9.451631	451.8817
20	434	439	444	439	5	10	429
30	437	433	436	435.3333	2.081666	4.163332	431.17
60	432	428	427	429	2.645751	5.291503	423.7085
120	435	425	426	428.6667	5.507571	11.01514	417.6515

**Note: all sample weights in mg; all temperatures in °C;  
 triplicate measurements were performed on each material;  
 \* samples disappeared after the tests without exhibiting an autoignition behavior**

Table V. Effect of sample quantity on the autoignition temperature of Nylon 6,6 at various oxygen pressures

**Effect of Sample Quantity on the AIT of Nylon 6,6 in 1800 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	209	211	209	209.6667	1.154701	2.309401	207.3573
10	207	205	203	205	2	4	201
20	202	201	202	201.6667	0.57735	1.154701	200.512
30	200	202	199	200.3333	1.527525	3.05505	197.2783
60	205	203	208	205.3333	2.516611	5.033223	200.3001
120	190	192	193	191.6667	1.527525	3.05505	188.6116
5	249	249	254	250.6667	2.886751	5.773503	244.8932
10	249	249	252	250	1.732051	3.464102	246.5359

**Effect of Sample Quantity on the AIT of Nylon 6,6 in 3000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	212	212	214	212.6667	1.154701	2.309401	210.3573
10	203	205	205	204.3333	1.154701	2.309401	202.0239
20	200	210	202	204	5.291503	10.58301	193.417
30	199	198	197	198	1	2	196
60	207	206	200	204.3333	3.785939	7.571878	196.7615
120	197	194	197	196	1.732051	3.464102	192.5359

**Effect of Sample Quantity on the AIT of Nylon 6,6 in 4000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	208	206	203	205.6667	2.516611	5.033223	200.6334
10	205	201	202	202.6667	2.081666	4.163332	198.5033
20	198	199	201	199.3333	1.527525	3.05505	196.2783
30	198	197	198	197.6667	0.57735	1.154701	196.512
60	197	198	195	196.6667	1.527525	3.05505	193.6116
120	194	191	192	192.3333	1.527525	3.05505	189.2783

**Effect of Sample Quantity on the AIT of Nylon 6,6 in 5000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	210	211	213	211.3333	1.527525	3.05505	208.2783
10	208	203	210	207	3.605551	7.211103	199.7889
20	205	199	200	201.3333	3.21455	6.429101	194.9042
30	199	197	197	197.6667	1.154701	2.309401	195.3573
60	199	200	196	198.3333	2.081666	4.163332	194.17
120	190	192	192	191.3333	1.154701	2.309401	189.0239

**Note:** all sample weights in mg; all temperatures in °C;  
 triplicate measurements were performed on each material

Table VI. Effect of sample quantity on the autoignition temperature of Vespel® SP-21 at various oxygen pressures

**Effect of Sample Quantity on the AIT of Vespel® SP-21 in 1800 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	390	395	382	389	6.557439	13.11488	375.8851
10	381	378	379	379.3333	1.527525	3.05505	376.2783
20	375	375	369	373	3.464102	6.928203	366.0718
30	365	369	375	369.6667	5.033223	10.06645	359.6002
60	367	364	372	367.6667	4.041452	8.082904	359.5838
120	370	363	363	365.3333	4.041452	8.082904	357.2504

**Effect of Sample Quantity on the AIT of Vespel® SP-21 in 3000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	383	380	379	380.6667	2.081666	4.163332	376.5033
10	368	365	361	364.6667	3.511885	7.023769	357.6429
20	356	352	358	355.3333	3.05505	6.110101	349.2232
30	357	353	356	355.3333	2.081666	4.163332	351.17
60	345	345	359	349.6667	8.082904	16.16581	333.5009
120	342	351	349	347.3333	4.725816	9.451631	337.8817

**Effect of Sample Quantity on the AIT of Vespel® SP-21 in 4000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	360	372	374	368.6667	7.571878	15.14376	353.5229
10	363	358	359	360	2.645751	5.291503	354.7085
20	358	354	353	355	2.645751	5.291503	349.7085
30	358	355	354	355.6667	2.081666	4.163332	351.5033
60	348	343	342	344.3333	3.21455	6.429101	337.9042
120	340	343	342	341.6667	1.527525	3.05505	338.6116

**Effect of Sample Quantity on the AIT of Vespel® SP-21 in 5000 psig Oxygen**

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
5	356	356	352	354.6667	2.309401	4.618802	350.0479
10	355	351	349	351.6667	3.05505	6.110101	345.5566
20	353	351	355	353	2	4	349
30	348	351	345	348	3	6	342
60	334	346	342	340.6667	6.110101	12.2202	328.4465
120	342	340	339	340.3333	1.527525	3.05505	337.2783

**Note: all sample weights in mg; all temperatures in °C;  
triplicate measurements were performed on each material**

# Effect of Oxygen Pressure and Sample Quantity on the AIT of Kel-F 81

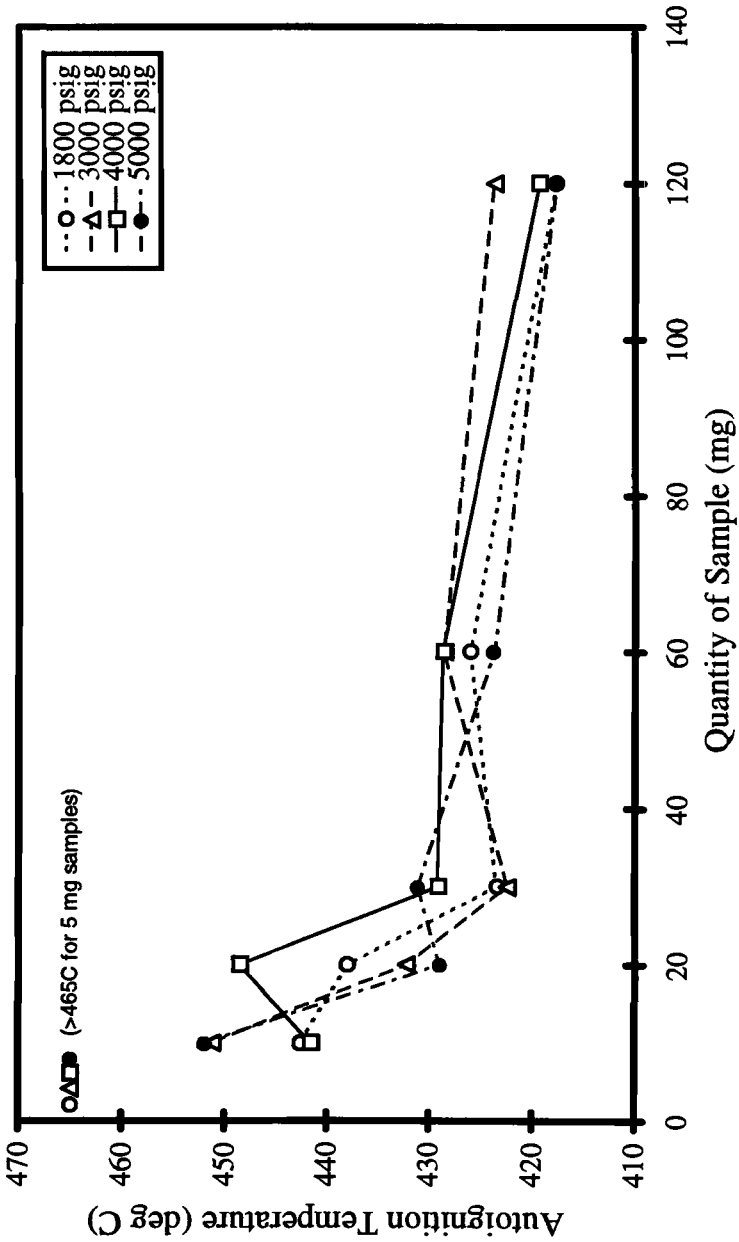


Fig.4 The effect of sample quantity on the autoignition temperature of Kel-F\* 81 at various oxygen pressures

# Effect of Oxygen Pressure and Sample Quantity on the AIT of Nylon 6,6

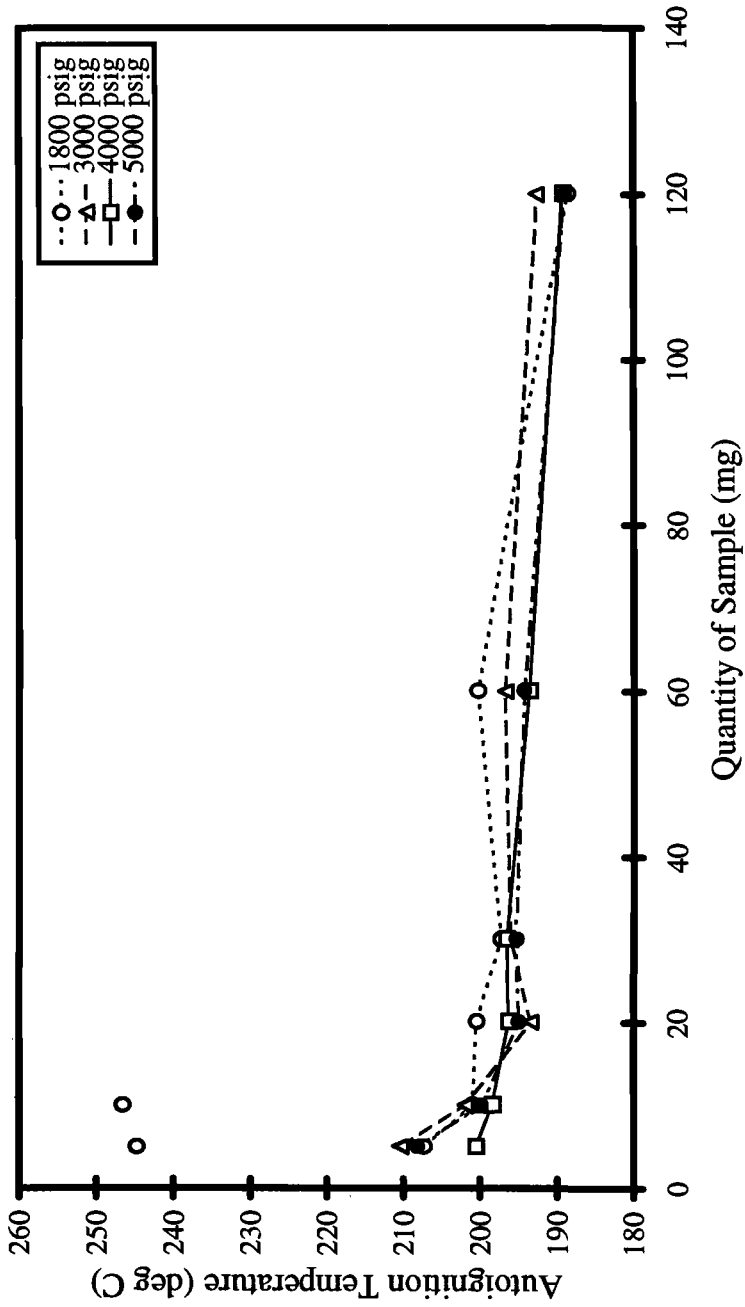


Fig.5 The effect of sample quantity on the autoignition temperature of Nylon 6,6 at various oxygen pressures

# Effect of Oxygen Pressure and Sample Quantity on the AIT of Vespel SP-21

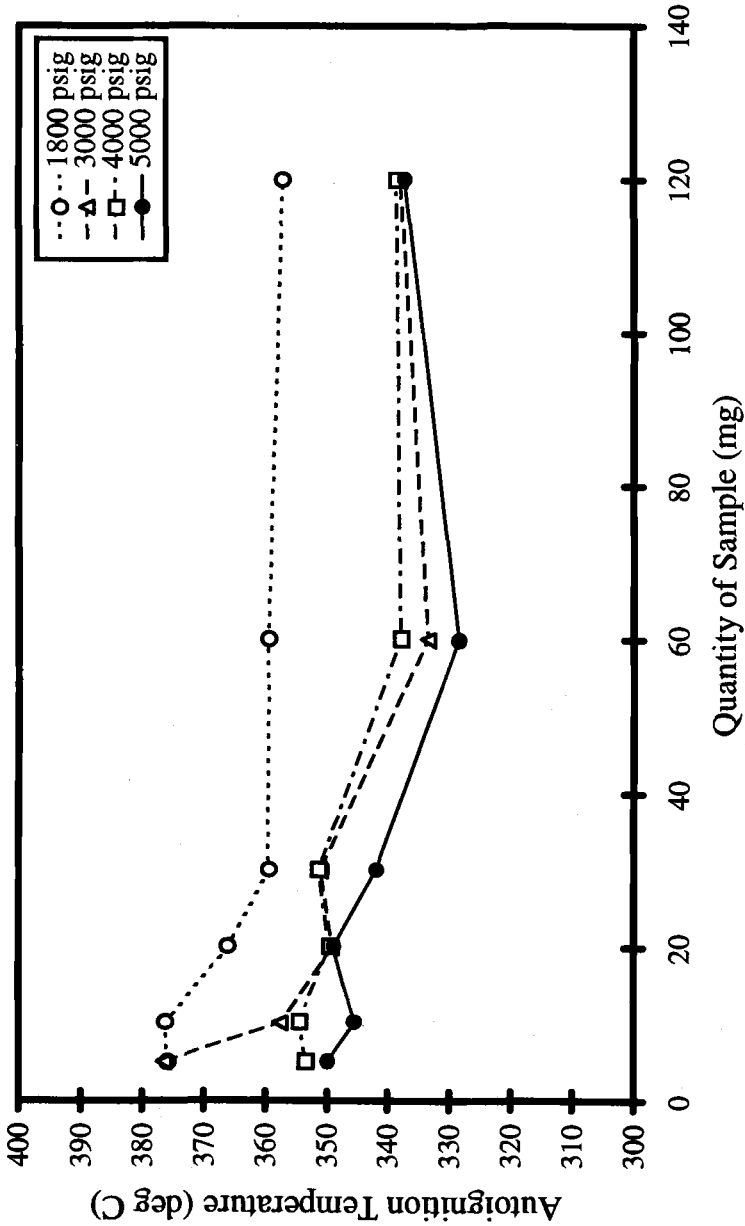


Fig.6 The effect of sample quantity on the autoignition temperature of Vespel® SP-21 at various oxygen pressures

### **(C) Autoignition Temperature vs. Sample Surface Area in 1800 psig (~12.4 Mpa) Oxygen**

The autoignition behavior of Kel-F® 81, Nylon 6,6, EPDM, Viton® A and Vespel® SP-21 in 1800 psig (~12.4 MPa) oxygen was further investigated as a function of sample surface area, by varying the number of test pieces dissected from a 60 mg sample. The experimental results are presented in Table VII and graphically illustrated in Fig.7. The autoignition temperatures of most materials including Kel-F® 81, Nylon 6,6, EPDM, and Viton® A showed negligible effect of sample surface area. By contrast, measurable changes in AITs were noted in Vespel® SP-21. Samples with the smallest surface area (i.e., 2 pieces per 60 mg in quantity) exhibited the highest AIT (387°C), which was about 25°C higher than that measured from samples with the largest surface area (i.e., 30 pieces per 60 mg in quantity).

## **II. HEAT OF COMBUSTION AND LOX MECHANICAL IMPACT SENSITIVITY**

Table VIII summarizes the heat of combustion value and LOX mechanical impact sensitivity of the polymeric materials. Rating of the materials in terms of heat of combustion can be expressed in a descending order as: TFE® > Kel-F® 81 > Viton® A-500/Viton® A/Fluorel® > Vespel® SP-21/Buna-N > Nylon 6,6/Neoprene® > EPDM. Since heat of combustion gauges the amount of thermal energy that is likely to be released upon the combustion of a material, it is recommended in principle that a material with a low heat of combustion value be selected for oxygen applications. According to ASTM guidelines [1], 2500 cal/gram was recommended as a demarcation line for the selection criterion. Based on the data shown in Table VIII, fluorinated polymers including TFE-Teflon®, Kel-F® 81, Viton® A-500, Viton® A and Fluorel® clearly stand up as promising candidates for oxygen application.

As for the materials' mechanical impact sensitivity to liquid oxygen, TFE-Teflon® outperformed the rest of the polymeric materials and was the only material that passed the LOX impact test. It is important to bear in mind that the mechanical impact energy employed by the BS 3N 100 LOX impact test is 122 joule, corresponding to energy per unit contact area 97.8 J/cm<sup>2</sup>. Such a level of impact energy is about 23% higher than that prescribed in the ASTM and CEN standard tests, which employ an impact energy 98 joule (corresponding to energy per unit contact area 79 J/cm<sup>2</sup>).



Table VII. Effect of sample surface area on the autoignition temperature of Kel-F® 81, Nylon 6,6, EPDM, Viton® A, and Vespel® SP-21 in 1800 psig oxygen

<u>Weight</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
<b>Effect of Sample Surface Area on the AIT of 60 mg Kel-F® 81 in 1800 psig Oxygen</b>							
<u># of Piece</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
2	420	418	418	418.6667	1.154701	2.309401	416.3573
6	420	420	418	419.3333	1.154701	2.309401	417.0239
12	420	420	418	419.3333	1.154701	2.309401	417.0239
20	418	418	418	418	0	0	418
30	418	419	419	418.6667	0.57735	1.154701	417.512
<b>Effect of Sample Surface Area on the AIT of 60 mg Nylon 6,6 in 1800 psig Oxygen</b>							
<u># of Piece</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
2	209	209	211	209.6667	1.154701	2.309401	207.3573
6	203	204	205	204	1	2	202
12	202	202	203	202.3333	0.57735	1.154701	201.1786
20	201	200	200	200.3333	0.57735	1.154701	199.1786
30	196	197	198	197	1	2	195
<b>Effect of Sample Surface Area on the AIT of 60 mg EPDM in 1800 psig Oxygen</b>							
<u># of Piece</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
2	226	224	222	224	2	4	220
6	224	224	223	223.6667	0.57735	1.154701	222.512
12	221	222	222	221.6667	0.57735	1.154701	220.512
20	224	222	221	222.3333	1.527525	3.05505	219.2783
30	223	222	223	222.6667	0.57735	1.154701	221.512
<b>Effect of Sample Surface Area on the AIT of 60 mg Viton® A in 1800 psig Oxygen</b>							
<u># of Piece</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
2	256	256	257	256.3333	0.57735	1.154701	255.1786
6	258	256	258	257.3333	1.154701	2.309401	255.0239
12	263	260	259	260.6667	2.081666	4.163332	256.5033
20	260	258	258	258.6667	1.154701	2.309401	256.3573
30	256	255	258	256.3333	1.527525	3.05505	253.2783
<b>Effect of Sample Surface Area on the AIT of 60 mg Vespel® SP-21 in 1800 psig Oxygen</b>							
<u># of Piece</u>	<u>Temp1</u>	<u>Temp2</u>	<u>Temp3</u>	<u>Average</u>	<u>Delta</u>	<u>2*Delta</u>	<u>AIT</u>
2	392	395	400	395.6667	4.041452	8.082904	387.5838
6	390	385	384	386.3333	3.21455	6.429101	379.9042
12	370	373	371	371.3333	1.527525	3.05505	368.2783
20	368	374	374	372	3.464102	6.928203	365.0718
30	365	369	368	367.3333	2.081666	4.163332	363.17

**Note: all temperatures in °C;  
triplicate measurements were performed on each material**

# Effect of Sample Surface Area on the AIT of Polymers in 1800 psig Oxygen

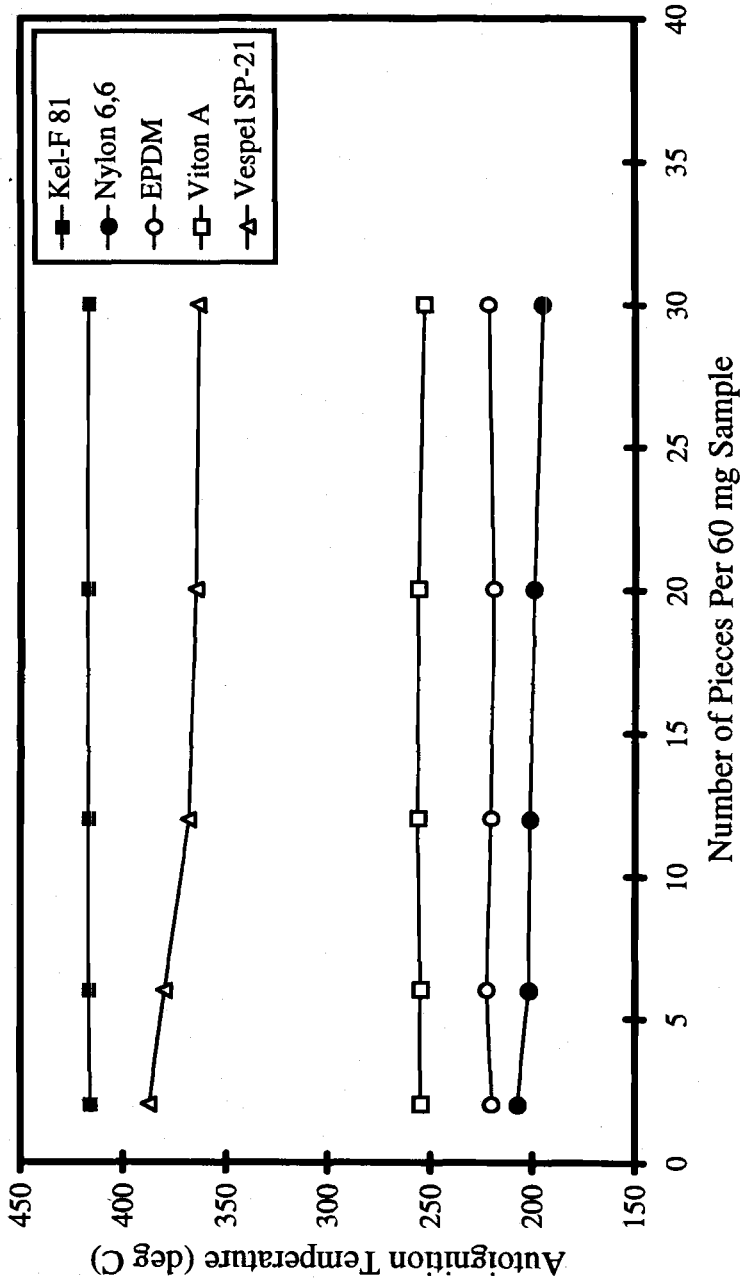


Fig. 7 The effect of sample surface area on the autoignition temperature of polymers in 1800 psig oxygen

Table VIII. Heat of combustion and liquid oxygen mechanical impact sensitivity of some polymeric materials

Property Material	ASTM D3286 Heat of Combustion (cal/gram)	BS 3N 100 LOX Impact Sensitivity (Pass/Fail)
TFE-Teflon° (PTFE)	<b>1517*</b> (1545/1505/1501)**	Pass
Kel-F° 81 (PCTFE)	<b>2556*</b> (2630/2644/2664)**	Fail
Vespel° SP-21	<b>6267*</b> (6266/6279/6256)**	Fail
Viton° A	<b>3992*</b> (4011/3975/3992)**	Fail
Viton° A-500	<b>3934*</b> (3945/3923/3936)**	Fail
Fluorel°	<b>3992*</b> (3983/3983/4012)**	Fail
Neoprene°	<b>7440*</b> (7451/7431/7440)**	Fail
EPDM	<b>9454*</b> (9481/9437/9445)**	Fail
Nylon 6,6	<b>7355*</b> (7349/7366/7351)**	Fail
Buna-N	<b>6271*</b> (6290/6267/6256)**	Fail

\* average value of heat of combustion

\*\* values obtained from triplicate measurements

## SUMMARY

According to the present study, the following conclusions can be reached:

(1) HP-AIT test results revealed that the oxygen compatibility of the polymeric materials could be ranked in a descending order as TFE-Teflon® (PTFE) > Kel-F® 81 > Vespel® SP-21 > Fluorel® > Viton® A/Viton® A-500 > EPDM > Nylon 6,6 > Neoprene®/Buna-N. Variation of oxygen pressure, ranging from 1800 psig (~12.4 MPa) to 5000 psig (~34.5 MPa), appeared to pose little effect on the autoignition temperatures of Kel-F® 81, Vespel® SP-21, Fluorel®, Viton® A/Viton® A-500, EPDM, Nylon 6,6, Neoprene®, and Buna-N.

(2) BS 3N 100 test results indicated that the polymeric materials could be rated as TFE-Teflon® (PTFE) > Kel-F® 81 > Vespel® SP-21 > Viton® A-500/Fluorel® > Viton® A > Neoprene® > EPDM > Nylon 6,6 > Buna-N.

(3) ASTM G-72 test results showed that the polymeric materials could be rated as TFE-Teflon® (PTFE) > Kel-F® 81 > Vespel® SP-21 > Fluorel® > Neoprene® > Viton® A/A-500 > EPDM > Nylon 6,6 > Buna-N.

(4) By comparing the data generated by ASTM and BSI vessels at their respective testing pressures, as well as by HP-AIT at 1800 psig (~12.4 MPa), ASTM vessel appeared to have produced the most conservative autoignition temperature, followed by BOC's HP-AIT vessel and then BSI vessel. An exceptional case, however, was noted for Neoprene whose AIT was the lowest according to HP-AIT tests, followed by the ASTM and BSI tests.

(5) Viton® A, Kel-F® 81, Vespel® SP-21, and Nylon 6,6 showed strong dependence of their autoignition behavior on the amount of sample quantity employed for the tests. Regardless of the pressure variation in oxygen, higher autoignition temperatures were consistently obtained from samples with smaller quantities, mostly less than 30 mg. Among all the materials studied, Viton A showed the most significant and consistent changes in its AITs. The largest variation in AITs was about 90°C.

(6) Kel-F® 81, Nylon 6,6, EPDM, and Viton® A exhibited a negligible effect of sample surface area on their autoignition behavior, although small but measurable changes in AITs were noted in Vespel SP-21. Samples with the smallest surface area (i.e., 2 pieces per 60 mg in quantity) exhibited the highest AIT (387°C), compared to that (363°C) measured from samples with the largest surface area (i.e., 30 pieces per 60 mg in quantity).

(7) Evaluations on the heat of combustion revealed that the polymeric materials could be ranked in a descending order as: TFE-Teflon® (PTFE) > Kel-F® 81 > Viton® A-500/Viton® A/Fluorel® > Vespel® SP-21/Buna-N > Nylon 6,6/Neoprene® > EPDM.

(8) Mechanical impact tests of the polymers in liquid oxygen, at atmospheric pressure and under 122 joules impact energy, indicated that TFE-Teflon® (PTFE) was the very only material passed the test without effecting an audible detonation or a visible ignition.

On the basis of the present study, TFE-Teflon® (PTFE), Kel-F® 81, Vespel® SP-21, and Viton® A/Viton® A-500/Fluorel exhibited consistently superior oxygen-compatible properties to the other polymers under the categories of autoignition temperature, heat of combustion, and LOX mechanical impact sensitivity. As a result, these materials are highly recommended for further prototype or live-component evaluations for compressed, high-pressure oxygen applications.

Since the oxygen compatibility of materials, particularly those polymer in nature, can be greatly influenced/alterd by processing additives (e.g., plasticizer and binding agent), a batch test should be performed on each procurement of a material to ensure the quality and consistency of its oxygen compatibility. Moreover, once a vendor/material supplier is identified for a specific quality product, adherence to the same for future supplies and requesting for material certificate(s) are paramount for quality assurance. Under situations where a less-oxygen compatible material is desirable for operation or performance reasons, it is advisable that the material be integrated as a functional component with the overall engineering structure and subjected to adiabatic compression tests and/or live component tests under the intended application conditions for final approval.

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