ISOTHERMAL WEIGHT LOSS AND LIFE EXPECTANCY CURVES OF
POLYMERS IN AIR

Polymer Branch
Nonmetallic Materials Division

June 1978

TECHNICAL REPORT AFML-TR-78-63

Final Report for Period May 1976 to January 1978

Approved for public release; distribution unlimited.
NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation wheresoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report has been reviewed by the Information Office (ASD/OIP) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

E. E. ARNOLD
Project Scientist

R. L. VAN DEUSEN, Chief
Polymer Branch
Nonmetallic Materials Division

FOR THE DIRECTOR

J. M. KELBLE, Chief
Nonmetallic Materials Division

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

AIR FORCE/56780/4 June 1979 — 125
**Title:** ISOTHERMAL WEIGHT LOSS AND LIFE EXPECTANCY CURVES OF POLYMERS IN AIR

**Author:** Gerhard F. L. Ehlers

**Performing Organization Name and Address:**
Air Force Materials Laboratory (AFML/MBP)
Wright-Patterson Air Force Base, OH 45433

**Report Date:** June 1978

**Number of Pages:** 1

**Abstract:**
Sixty-five polymers have been subjected to isothermal aging in air for up to 200 hours at one or several temperatures of 500, 550, 600, 650, 700, and 750°F and the weight loss-time curves plotted. The data have been used to obtain life expectancy plots for 20 and for 30% weight loss.
FOREWORD

This report was prepared by the Polymer Branch, Nonmetallic Materials Division. The work was initiated under WUD #43, "Structural Resins". It was administered under the direction of the Air Force Materials Laboratory, Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with Dr. G. F. L. Ehlers (AFML/MBP) as Project Scientist.

This report covers work conducted from May 1974 to January 1978. It was submitted for publication by the author in February 1978.
TABLE OF CONTENTS

SECTION                        PAGE
----------------------------------------
I      INTRODUCTION                  1
II     DISCUSSION                    2
III    WEIGHT LOSS CURVES            3
IV     LIFE EXPECTANCY CURVES        69
# LIST OF WEIGHT LOSS CURVES

<table>
<thead>
<tr>
<th>Polymer Class</th>
<th>Pages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluoroaliphatic Polymers</td>
<td>4 - 5</td>
</tr>
<tr>
<td>Epoxy Resin</td>
<td>6</td>
</tr>
<tr>
<td>Phenolic Resin</td>
<td>7</td>
</tr>
<tr>
<td>Polyphenylenes</td>
<td>8 - 10</td>
</tr>
<tr>
<td>Polyphenylene oxides</td>
<td>11 - 16</td>
</tr>
<tr>
<td>Polyphenylene sulfones</td>
<td>15 - 16</td>
</tr>
<tr>
<td>Polyphenylene sulfide</td>
<td>17</td>
</tr>
<tr>
<td>Aromatic Polyesters</td>
<td>18 - 19</td>
</tr>
<tr>
<td>Aromatic Polycarbonate</td>
<td>20</td>
</tr>
<tr>
<td>Aromatic Polyamides</td>
<td>21 - 24</td>
</tr>
<tr>
<td>Polymides</td>
<td>25 - 30</td>
</tr>
<tr>
<td>Polybenzimidazole</td>
<td>31 - 35</td>
</tr>
<tr>
<td>Polybenzoxazoles</td>
<td>36 - 42</td>
</tr>
<tr>
<td>Polyoxadiazoles</td>
<td>43 - 44</td>
</tr>
<tr>
<td>Polybenzothiazoles</td>
<td>45 - 46</td>
</tr>
<tr>
<td>Polythiadiazone</td>
<td>47</td>
</tr>
<tr>
<td>Polyquinoxalines</td>
<td>48 - 57</td>
</tr>
<tr>
<td>BBB and related structures</td>
<td>58 - 63</td>
</tr>
<tr>
<td>Miscellaneous heterocyclic polymers</td>
<td>64 - 66</td>
</tr>
<tr>
<td>Silicon containing polymers</td>
<td>67 - 68</td>
</tr>
</tbody>
</table>
# List of Life Expectancy Curves

<table>
<thead>
<tr>
<th>Polymer Class</th>
<th>Pages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyphenylenes</td>
<td>70 - 71</td>
</tr>
<tr>
<td>Polyphenylene oxides</td>
<td>72 - 75</td>
</tr>
<tr>
<td>Polyphenylene sulfide</td>
<td>76</td>
</tr>
<tr>
<td>Aromatic polyester</td>
<td>77</td>
</tr>
<tr>
<td>Aromatic polycarbonate</td>
<td>78</td>
</tr>
<tr>
<td>Aromatic polyamides</td>
<td>79 - 80</td>
</tr>
<tr>
<td>Polyimides</td>
<td>81 - 83</td>
</tr>
<tr>
<td>Polybenzimidazoles</td>
<td>84 - 88</td>
</tr>
<tr>
<td>Polybenzoxazoles</td>
<td>89 - 90</td>
</tr>
<tr>
<td>Polyoxadiazole</td>
<td>91</td>
</tr>
<tr>
<td>Polythiadiazole</td>
<td>92</td>
</tr>
<tr>
<td>Polyquinoxalines</td>
<td>93 - 97</td>
</tr>
<tr>
<td>Polymers related to BBB</td>
<td>98 - 100</td>
</tr>
<tr>
<td>Composite Life Expectancy Curves,</td>
<td></td>
</tr>
<tr>
<td>based on 20% weight loss</td>
<td>101</td>
</tr>
<tr>
<td>Composite Life Expectancy Curves,</td>
<td></td>
</tr>
<tr>
<td>based on 30% weight loss</td>
<td>102</td>
</tr>
</tbody>
</table>
SECTION I
INTRODUCTION

The development of an automatic multisample isothermal aging apparatus (AFML-TR-74-163) allowed the simultaneous thermal aging of up to four polymer samples in duplicate.

Since then, a representative variety of polymers has been subjected to isothermal aging at different temperatures. The weight loss plots and some life expectancy curves derived from these plots are presented in this report.
Sixty-five polymers have been subjected to isothermal aging in air at one or several temperatures of 500, 550, 600, 650, 700, and 750°F for up to 200 hours, in one case (BBB) up to 600 hours. Aging was performed in a Multisample Isothermal Aging Apparatus designed and built by the Systems Research Laboratories in cooperation with Polymer Branch personnel. Sample sizes of about 20 mg, in aluminum boats, were heated in an air flow of 40 CC/min. The curves shown in this report are average plots from two or more samples. Many of the polymers show an inhibition period of several hours up to 150 (BBB) after which the decomposition accelerates.

For 31 of these polymers, life expectancy plots were made. Basis for these plots was the elapsed time after the samples reached weight losses of 20 and 30%. Weight losses in this order would still be acceptable in cases where, for example, laminates are being used as separators, dielectric barriers, etc. In a laminate based on glass fabric and a resin content of 35%, a 30% weight loss would mean a reduction of the resin content to 25%.

Some of the life expectancy plots are of a convex, others of a concave shape. A satisfactory explanation, why these differences occur, has not been found. As it turns out, structurally related polymers have differently shaped life curves; ATQ-s and ATQ-p have convex life expectancy curves, the one of ATQ-m.p. appears to be a straight line, and the ATQ-o curve is concave.

Composite plots of all of the life expectancy curves have been made, separately for 20 and for 30% weight loss. The composite plots indicate that most of the curves (all of these are from aromatic or aromatic-heterocyclic polymers) fall in the same range.
SECTION III
WEIGHT LOSS CURVES
Weight Residue (%) vs. Time (hours)

500°F

600°F

\[
\text{VITON A} \quad \left( -\frac{\text{CF}_2}{\text{CF}_3} \right)_n \left( -\frac{\text{CF}_2}{\text{CH}_2} \right)_m
\]
DU PONT 006E

\((-\text{CF}_2 - \text{CF}_2\) \_m \((-\text{CF}_2 - \text{CF} - \text{CF}_3\)_n

\sim 70\% \text{ m/m} \quad \sim 30\%
Trm P105a
Polyimide from Nadic Anhydride,
Benzophenone Dicarboxylic Acid
and Methylene Dianiline

Weight Residue (%)

Time (hours)

650°F

700°F
Weight Residue (%) vs. Time (hours)

- **PPB0-108**: $R_1 = H; R_2, R_3, R_4 = -C_6H_5$
- **PPB0-114**: $R_1, R_2, R_3, R_4 = -C_6H_5$
- **PPB0-116**: $R_1, R_2 = H; R_3, R_4 = -C_6H_5$

Chemical structure of the molecule:

![Chemical structure diagram]
ATQ-MP (cured 8 hrs 280°C)
The image shows a graph with two lines representing different temperatures: 650°F and 700°F. The x-axis represents weight residue (%) while the y-axis represents time (hours) ranging from 0 to 200. A molecular structure is also present on the right side of the graph.
SECTION IV
LIFE EXPECTANCY CURVES
FIBER B

\[
\begin{align*}
&O \\
&C_6H_4 &\text{O} &\text{H} \\
&\text{C-N} &\text{C-N} \\
&\text{NH} &\text{NH} \\
&\text{[n]} \\
\end{align*}
\]

Life Expectancy (Hours)

\begin{align*}
\text{Life Expectancy} & = \text{Function of } \frac{1}{T} \times 10^{-3} (\text{°K}) \\
\end{align*}

- 30% mark
- 20% mark

Temperature (°C)

- 750
- 700
- 650
- 600
- 550
- 500

1/T x 10^{-3} (°K)

- 1.0
- 1.1
- 1.2
- 1.3

AFML-TR-78-63
TRW P13N
assumed to be:

\[
\begin{align*}
\text{Life Expectancy (Hours)} \\
1/T \times 10^{-3} \, (^\circ K)
\end{align*}
\]
TRW P105A
assumed to be:
Polyimide from Nodic Anhydride,
Benzophenone Dicarboxylic Acid
and Methylene Dianiline

1/T x 10^{-3} (°K)

750 700 650 °C 600 550 500

1.0 1.1 1.2 1.3

Life Expectancy (hours)

3 2

1 10

10

100

1000

30%

20%
Life Expectancy (hours)

1/T x 10^{-3} (°K)

750 700 650 °C 600 550 500

30% 20%

[Chemical Structure]
Life Expectancy (Hours)

\[ \frac{1}{T} \times 10^{-3} (\text{°K}) \]

\[ 750 \quad 700 \quad 650 \quad 600 \quad 550 \quad 500 \]

30% 20%

NCF20 F 50 F)2-CC -(CF2)4(CF20 F n
ATQ-o
(cured 8 hrs 280°C)
Composite Life Expectancy Curves,
Based on 20% Weight Loss

Life Expectancy (Hours)

1/T x 10^-3 (°K)

750 700 650 °C 600 550 500
Composite Life Expectancy Curves, Based on 30% Weight Loss