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COMPARISON OF THE WEIGHT LOSS AND ADHERENCE OF NINE DIFFERENT POLYIMIDE FILMS THERMALLY AGED AT 315° AND 350° C IN AIR

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COMPARISON OF THE WEIGHT LOSS AND ADHERENCE OF 9 DIFFERENT POLYIMIDE FILMS THERMALLY AGED AT 315° C AND 350° C IN AIR

Robert L. Fusaro

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SUMMARY

Thermal exposure experiments were conducted at 315° C and 350° C to determine which of nine different types (chemical structures) of polymides were the most thermally stable and adherent when subjected to long exposure times at elevated temperatures. The films (of various weights and thicknesses) were applied to 304 stainless steel foils of dimensions 6.3 cm by 5.1 cm by 0.015 cm, and exposed in air for time durations up to 766 hours at 315° C and up to 213 hour at 350° C. It was found that the rate of film weight loss was constant with time for both exposure temperatures, and as film weight increased so did the rate of film weight loss. Thus the weight loss was more bulk oriented than surface oriented, since surface area was constant. The weight loss rate at 350° C was found to be about 5 times the weight loss rate at 315° C for all polyimide types.

The polyimides were ranked according to the rate at which they lost weight, from best to worst. It was determined that there were five groups, one polyimide, designated PIC-7, was found to be the most thermally stable, therefore it was ranked in group 1. In group 2 are PIC-2, PIC-3, PIC-5, and PMR-II; in group 3 is PIC-1; in group 4 is PIC-4 and PIC-6 and in group 5 is PMR-15.

Even though polyimide type PIC-7 was the most thermally stable, its adherent properties for long high temperture exposures were not as good as PIC-5 and PIC-2. As far as adherence was concerned, three different groups were ascertained. In the first group were PIC-5 and PIC-2; in the second group were PIC-3, PIC-7, and PMR II; and in the third group were PIC-1, PIC-4, PIC-6, and PMR-15. The adherence of group 3 was very poor.

INTRODUCTION

It has been shown in previous work (refs. 1 and 2) that polyimide-bonded graphite fluoride films have potential for solid lubricant applications where long thermal soaks are prevalent. Low weight loss, good adhesion, and good friction and wear properties were found for films thermally aged at temperatures up to 315° C.

Polyimide is a generic designation and refers to a class of long chain polymers which have recurring imide groups as an integral part of the main chain. By varying the monomeric starting materials, polyimides of different chemical composition and structure can be obtained.

N80-18183

In previous friction and wear studies (refs. 1 to 9) a commercially available polyimide designated PI-4701 was used. There are now several other commercial polyimides available. In addition, two promising polyimides have been developed at NASA-Lewis Research Center (refs. 10 to 14).

The objective of this investigation was to determine which of those polyimides was the most thermally stable and most adherent at elevated temperatures.

Seven different commercially available polyimides were avaluated, they were designated as PIC-1 to PIC-7. PIC-1 was the polyimide used in previous investigations (refs 1 to 9). Two NASA polyimides were also evaluated which were designated PMR-15 and PMR II. The films were exposed at temperatures of 315° C (599° F) or 350° C (662° F) and weight loss and adherence were determined for various exposure times.

MATERIALS

The polyimides used were obtained as precursor solutions. In most instances a thinner consisting of N-methyl-pyrrolidone and xylene was added to the polyimides to make them sprayable. Nine different types of polyimides were evaluated, seven condensation-type polyimides and two addition-type polyimides. The chemical composition and structure of 5 of the condensation polyimides were proprietary. They are designated in table I as polyimide types PIC-1 to PIC-5. The structure of the other two polyimides was known and are shown in figures 1 and 2. They are designated as polyimide types PIC-6 and 7 in this investigation.

The additive-type polyimides were developed at NASA-Lewis Research Center (refs. 10 to 14). They are designated as PMR-15 and PMR II. The monomers used in the synthesis of these polyimides are shown in tables II and III.

The polyimide which was used previously by this investigator is designated in this study as PIC-1.

The films were applied to 304 stainless steel foils (6.3 by 5.1 by 0.015 cm) that had a hardness of Rockwell B-87.

PROCEDURE

Surface Preparation and Cleaning

The foil surfaces were roughened by sanding with number 150 wet sand paper to a cla roughness of 0.25 to 0.36 micrometer. After surface roughening, the foils were scrubbed with a brush under running tap water to ensure that no abrasive particles remained. A water paste of levigated alumina was next rubbed by hand over the surface with a polishing cloth. This was followed by a second scrubbing under running tap water. The foils

were rinsed in distilled water and clean, dry compressed air was then used to quickly dry the surfaces. The foils were stored in a desiccator until they were coated with the solid lubricant.

Film Application and Cure

An artist's airbrush was used to apply the polyimide films to the foils. The films did not dry rapidly; thus, only a thin layer was applied at one time in order to prevent "running". Each layer was completely cured before the next layer was applied. The cure was to heat from 25° C to 75° C in 1/2 hour, heat from 75° C to 125° C in 1/2 hour, heat from 125° to 175° C in 1/2 hour, heat from 175° C to 225° C in 1/2 hour, and heat from 225° C to 300° C in 3/4 hour then hold at 300° C for 1 hour. The films were weighed after each cure to determine if another layer was necessary to achieve the desired weight.

The polyimides used in this study are not normally applied as thin films, thus the curing procedure (as given by the manufacturer) was not fully specified. The basic curing theory is to drive the H_2O and solvent out of the pre cursor solution at a low temperature before polyimerization takes place at a higher temperature. Polyimerization is time and temperature dependent. The higher the temperature, the shorter the time needed to achieve the same degree of polyimerization. In previous studies on PIC-1 polyimides (refs. 1-9), the cure procedure was to bake at 100° C for 1 hour and at 300° C for 2 hours. This cure did not work on PIC-7 polyimide; the films were found to be cracked and blistered. To obtain a good film with PIC-7 polyimide, it was necessary to raise the temperature more slowly from room temperature. Thus the previously mentioned cure schedule was devised to obtain good PIC-7 films. The same cure was tried on the six other condensation polyimides and good films were also obtained. Therefore the previously mentioned cure schedule was used as the standard in this study.

Thermal Aging Tests

The thermal aging tests were conducted at temperatures of 315° C (599° F) and 350° C (662° F). At various intervals they were taken out of the oven, let cool for 15 minutes, and weighed to obtain a weight loss. Cooling time was kept to a minimum, so such variables as water absorption could be kept to a minimum. The foils were aged for as long as 766 hours at 315° C and 213 hours at 350° C.

Adherence Tests

The adherence tests were performed on the films applied to the foils before and after thermal aging. They consisted of simple bending and flexing tests of the foils. The foils with applied films were repeatedly flexed and bent by hand to various degrees and then observed under optical

microscopy for cracking. In many instances thermal exposure alone caused the films to spall.

RESULTS AND DISCUSSION

Film weight loss at 315° C

Nine different types of polyimides (table I) were thermally exposed in air to temperatures of 315° C for time intervals of up to 766 hours. In addition to comparing the rate of weight loss of the various polyimides, the effect of total film weight on the amount of weight loss was also investigated. The weight loss rates of at least 4 different film weights for each polyimide were determined.

Figures 3 to 10 give polyimide film weight losses at 315° C as a function of exposure time for the nine different types of polyimides. The figures illustrate that the rate of film weight loss was constant for all the films regardless of film weight.

A linear regression fit (least squares) of the film weight loss data points (for each exposure test) was made to obtain weight loss rates. Table IV compares these film weight loss rates for the nine polyimide films relative to film weight ranges of 0.0100 gm. As a general rule, the heavier the film, the greater the film weight loss rate.

Table V compares the same data of table IV, but in terms of precent film weight loss per hour. When looked at this way, it is seen that the percent of the film lost per hour is either constant or decreases slightly as a function of increasing weight. Thus it appears the weight loss of polyimide films is bulk oriented as well as surface oriented, since the surface area was constant.

The fact that most of the weight loss curves of figures 3 to 10 do not pass through zero of the y-axis indicates a considerable amount of water and or other volatile constituent was liberated from the films during initial heating at 315° C. In general, the heavier the film, the greater the weight loss. For most of the films, the water (or other volatile constituent) tended to be released in less than 24 hours of exposure; but for polyimide type PIC-5, it took up to 100 hours.

Water vapor absorption of the polyimide films was so intense that the foils could not be weighed as they were taken from the oven. The films constantly gained weight and a reading could not be made. It was found that by waiting 15 minutes before weighing that the absorption rate had decreased enough to allow a stable reading to be made. Therefore a standard waiting period of 15 minutes in room air was employed between oven removal and weighing of the specimens.

Film Weight Loss at 350°

Only the seven condensation polyimides were evaluated at 350° C. Figures 11 to 17 give the weight loss of these polyimides as a function of exposure time at 350° C. As found for exposure temperatures of 315° C, the rate of film weight loss was constant for all polyimides.

Table VI compares the film weight loss rates in terms of the film weight lost per hour, and table VII compares film weight loss in terms of percent film weight lost per hour. As found for the films exposed to 315° C, the heavier the film, the greater the amount of weight lost; but the percent of film weight lost per hour either decreased or remained the same. This again implies the weight loss is bulk oriented as well as surface oriented.

As found for the 315° C exposure tests, many of the weight loss curves for exposure temperatures of 350° C (figs. 11 to 17) do not pass through the zero of the y-axis, which again indicates a considerable amount of H_2O (or other volatile constituent) was liberated during initial exposure. In some cases, the rate of H_2O (or other volatile constituent) weight loss was small compared to the polyimide film weight loss and thus the curves tend to pass through zero for those polyimides.

Comparison of Weight Loss Rates

Table VIII compares the percent weight loss rates for the nine different polyimides evaluated at 315° C and 350° C. For all the polyimides the weight loss rate at 350° C was about 5 times greater than at 315° C. But the relative ranking of the polyimides, as far as thermal stability is concerned, is the same at 315° C and 350° C.

According to thermal stability, the polyimides might be divided into five groups. In group 1, there is one polyimide: PIC-7. This polyimide was the most thermally stable. The weight loss at 315° C was 0.025 $\pm$ 0.009 percent per hour and at 350° C was 0.13 $\pm$ 0.04 percent per hour. In group 2, the second most thermally stable polyimides, there are 4 polyimides: PIC-2, PIC-3, PIC-5, and PMR-II, at 315° C, the weight loss rates were: PIC-2, 0.042 $\pm$ 0.011; PIC-3, 0.042 $\pm$ 0.017; PIC-5, 0.043 $\pm$ 0.014; and PMR-II, 0.036 percent/hour. (Only one PMR film was available for evaluation). At 350° C, the weight loss rates were: PIC-2, 0.20 $\pm$ 0.07; PIC-3, 0.20 $\pm$ 0.10, and PIC-5 0.27 $\pm$ 0.08 percent/hour. PMR II was not evaluated at 350° C.

In group 3 there is only one polyimide: PIC-1. This is the polyimide that has been used in previous studies (refs. 1 to 9). The weight loss of PIC-1 at 315° C was 0.053 $\pm$ 0.014 percent/hour while at 350° C it was 0.26 $\pm$ 0.03 percent/hour.

In group 4 are PIC-4 and PIC-6. The weight loss of PIC-4 was 0.087 $\pm$ 0.015 percent/hour at 315° C and 0.34 $\pm$ 0.03 percent/hour at 350° C.

The weight loss of PIC-6 at 315° C was 0.079 ± 0.030 percent/hour and at 350° C was 0.40 ± 0.15 percent/hour.

The film which gave the largest weight loss was PMR-15 and might be considered as group 5. The weight loss at 315° C was 0.175 percent per hour. Only one PMR-15 film was available for evaluation.

Adherence of the Films

The adherence of the films was evaluated by simple bending tests both before and after thermal exposure to temperatures of 315° C and 350° C. The bending tests did not cause the films to spall; however the long thermal soaks did cause some of the films to blister and spall, both at 315° C and 350° C.

Table IX gives the exposure times when cracking and spalling of the films occurred at 315° C and table X gives the same information for 350° C. No cracking or spalling at 315° C was observed for 5 of the films: PIC-2, PIC-3, PIC-5, PIC-7, and PMR II. The other 4 showed some form of cracking or spalling. The trend was that the thicker the film, the sooner these cracks and spalls occurred.

At 350° C, cracks were found to occur in each type of polyimide; however in some individual cases no cracks were found. The most adherent polyimide at 350° C was PIC-5. Of the six, PIC-5 films evaluated at 350° C, only a small amount of cracking was observed in one corner of one foil after 184 hours of exposure. The next best was PIC-2. Of the six PIC-2 foils evaluated at 350° C only two showed fine cracking. No spalls were observed to occur in either PIC-5 or PIC-2.

Even though PIC-7 gave the lowest weight loss rates, it was not the best as far as adherence was concerned. Only one of the four PIC-7 films evaluated at 350° C did not crack or spall and that was the thinnest film. Equivalent results were found with PIC-3. Two of the four PIC-3 films evaluated at 350° C cracked and spalled. The other three polyimides evaluated, PIC-1, PIC-4, and PIC-6 all experienced severe cracking and spalling at 350° C, and are not considered good candidates for use when long thermal soaks are prevalent.

CONCLUDING REMARKS

Nine different polyimides were evaluated at 315° C and 350° C to determine which were the most thermally stable and which were the most adherent after long duration high temperature soaks. It was concluded the polyimides could be arranged into five different weight loss groups and three different adhesion groups. Table XI classifies the polyimides as to groups.

From the weight loss and adherence considerations, it was concluded that the polyimides of groups 1 and 2 (from both weight loss and adherence

categories) are the most suitable for high temperature applications. That is, polyimide types PIC-2, PIC-3, PIC-5, PIC-7, and PMR-II (not necessarily in that order) are the most promising.

SUMMARY OF RESULTS

Thermal exposure experiments at 315° C and 350° C on nine different types of polyimides indicate that:

1. The polyimides can be arranged into five different groups (from best to worst) relative to weight loss rates.

   (a) Group 1 PIC-7
   (b) Group 2 PIC-2, PIC-3, PIC-5, PMR-II
   (c) Group 3 PIC-1
   (d) Group 4 PIC-4, PIC-6
   (e) Group 5 PMR-15

2. The polyimides can be arranged into three different groups as far as adherence is concerned (from best to worst).

   (a) Group 1 PIC-5. PIC-2
   (b) Group 2 PIC-3, PIC-7, PMR II
   (c) Group 3 PIC-1, PIC-4, PIC-6, PMR-15

3. Weight loss rates were found to be constant with exposure time and the rate of weight loss increased about 5 times in going from 315° C to 350° C.

4. All polyimide films were found to contain large amounts of water vaper which was released on thermal exposure to 315° C or 350° C. It was also found that water vapor rapidly reabsorbed on cooling.

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TABLE I. - DESIGNATION OF TYPES OF POLYIMIDES

USED IN THIS INVESTIGATION

Designation	Type	Structure
PIC-1	Condensation	Proprietary
PIC-2	Condensation	Proprietary
PIC-3	Condensation	Proprietary
PIC-4	Condensation	Proprietary
PIC-5	Condensation	Proprietary
PIC-6	Condensation	Fig. 1
PIC-7	Condensation	Fig. 2
PMR-15	Addition	Table II
PMR-II	Addition	Table III

TABLE II. - MONOMERS USED FOR PMR-15 SYNTHESIS

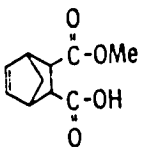
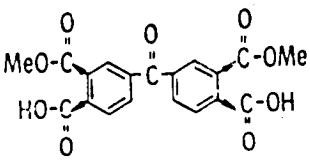
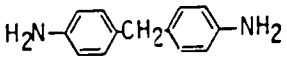
Structure	Name	Abbreviation
	Monomethyl ester of 5-norbornene - 2, 3-dicarboxylic acid	NE
	Dimethyl ester of 3, 3', 4, 4'-benzophenone - tetracarboxylic acid	BTDE
	4, 4'-Methylenedianiline	MDA

TABLE III. - MONOMERS USED FOR PMR-II SYNTHESIS

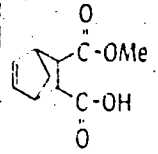
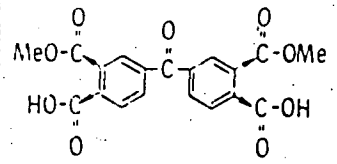
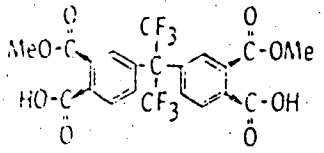
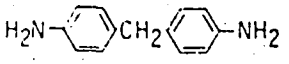
Structure	Name	Abbreviation
	Monomethyl ester of 5-norbornene-2, 3-dicarboxylic acid	NE
	Dimethyl ester of 3, 3', 4, 4'-benzophenone-tetracarboxylic acid	BTDE
	Dimethyl ester of 4, 4'-(hexafluoroisopropylidene)-bis (phthalic acid)	HFDE
	4, 4'-Methylenedianiline	MDA

TABLE IV. - COMPARISON OF THE AMOUNT OF POLYIMIDE FILM WEIGHT LOST PER HOUR AT 315° C FOR NINE DIFFERENT TYPES OF POLYIMIDES OF VARIOUS WEIGHTS

Film weight, g	Rate of film weight loss, microgram/hour								
	PIC-1	PIC-2	PIC-3	PIC-4	PIC-5	PIC-6	PIC-7	PMR-15	PMR-II
<0.0300	--	14	--	15	--	30	--	--	--
0.0300 to 0.0399	18 --	16 19	21	23	16	--	6	--	--
0.0400 to 0.0499	20	21	21	--	--	--	--	--	--
0.5000 to 0.0599	-- -- --	-- -- --	21 -- --	-- -- --	28 29 --	-- -- --	14 10 12	-- -- --	-- -- --
0.0600 to 0.0699	42	--	16	--	22	--	--	--	--
0.0700 to 0.799	-- --	26 25	41 --	-- --	23 --	64 --	15 --	-- --	-- --
0.0800 to 0.0899	-- --	26 29	-- --	84 --	-- --	-- --	28 --	-- --	-- --
0.0900 to 0.0999	40 --	29 --	-- --	-- --	-- --	83 83	-- --	-- --	-- --
0.1000 to 0.1099	--	--	--	--	--	--	--	--	--
0.1100 to 0.1199	--	--	--	--	--	56	--	--	--
0.1200 to 0.1299	49	--	--	--	--	--	20	--	--
0.1300 to 0.1399	--	--	--	--	--	--	--	--	--
0.1400 to 0.1299	--	--	--	--	--	98	--	--	--
>0.1500	--	--	--	158	--	--	--	263	55

TABLE V. - COMPARISON OF THE PERCENT OF POLYIMIDE FILM WEIGHT LOST PER HOUR AT 315° C FOR NINE DIFFERENT TYPES OF POLYIMIDE OF VARIOUS WEIGHTS

Film weight, g	Rate of weight loss, percent/hour								
	PIC-1	PIC-2	PIC-3	PIC-4	PIC-5	PIC-6	PIC-7	PMR-15	PMR-II
<0.0300	--	0.048	--	0.072	--	0.109	--	--	--
0.0300 to 0.0399	0.053	0.053	0.058	0.073	0.040	--	0.016	--	--
	--	0.053	--	--	--	--	--	--	--
0.0400 to 0.0499	0.050	0.053	0.042	--	--	--	--	--	--
0.0500 to 0.0599	--	--	0.040	--	0.057	--	0.027	--	--
	--	--	--	--	0.054	--	0.018	--	--
	--	--	--	--	--	--	0.024	--	--
0.0600 to 0.0699	0.066	--	0.025	--	--	--	--	--	--
0.0700 to 0.0799	--	0.036	0.059	--	0.032	0.081	0.021	--	--
	--	0.032	--	--	--	--	--	--	--
0.0800 to 0.899	--	0.031	--	0.097	0.030	--	0.034	--	--
	--	0.032	--	--	--	--	--	--	--
0.0900 to 0.0999	0.042	0.032	--	--	--	0.088	--	--	--
	--	--	--	--	--	0.084	--	--	--
0.1000 to 0.1099	--	--	--	--	--	--	--	--	--
0.1100 to 0.1199	--	--	--	--	--	0.049	--	--	--
0.1200 to 0.1299	0.039	--	--	--	--	--	0.016	--	--
0.1300 to 0.1399	--	--	--	--	--	--	--	--	--
0.1400 to 0.1499	--	--	--	--	--	0.069	--	--	--
>0.1500	--	--	--	0.102	--	--	--	0.175	0.036

TABLE VI -COMPARISON OF THE PERCENT OF POLYIMIDE FILM WEIGHT LOST PER HOUR AT 350° C FOR SEVEN DIFFERENT TYPES OF POLYIMIDES OF VARIOUS WEIGHTS

Film weight g,	Rate of film weight loss, percent/hour								
	PIC-1	PIC-2	PIC-3	PIC-4	PIC-5	PIC-6	PIC-7	PMR-15	PMR-II
<0.0300	--	--	--	--	--	--	--	--	--
0.0300 to 0.0399	--	0.27	--	--	0.35	--	0.17	--	--
	--	0.23	--	--	0.33	--	--	--	--
0.0400 to 0.499	--	0.20	--	--	0.20	--	--	--	--
	--	--	--	--	0.21	--	--	--	--
0.0500 to 0.0599	--	0.20	0.30	--	--	--	--	--	--
0.0600 to 0.699	0.29	--	0.26	0.37	0.28	0.55	--	--	--
	0.29	--	--	--	--	--	--	--	--
0.0700 to 0.799	--	0.14	0.23	--	--	0.50	0.09	--	--
0.0800 to 0.899	--	--	--	0.32	--	--	--	--	--
0.0900 to 0.0999	--	--	--	--	--	--	--	--	--
0.1000 to 0.1099	--	--	--	--	--	--	0.11	--	--
0.1100 to 0.1199	0.24	--	--	--	--	0.24	--	--	--
0.1200 to 0.1299	--	0.12	--	--	--	0.036	0.12	--	--
0.1300 to 0.1299	--	--	0.19	--	--	0.25	--	--	--
0.1400 to 0.1499	--	--	--	--	--	--	--	--	--
>0.1500	0.23	--	--	--	--	--	--	--	--

TABLE VII. - COMPARISON OF THE AMOUNT OF POLYIMIDE FILM WEIGHT LOST PER HOUR AT 350° C FOR SEVEN DIFFERENT TYPES OF POLYIMIDES OF VARIOUS WEIGHTS

Film weight, g,	Rate of film weight loss, microgram/hour								
	PIC-1	PIC-2	PIC-3	PIC-4	PIC-5	PIC-6	PIC-7	PMR-15	PMR-II
<0.0300	--	--	--	--	--	--	--	--	--
0.0300 to 0.0399	-- --	90 86	-- --	-- --	120 120	-- --	60 --	-- --	-- --
0.400 to 0.0499	-- --	86 --	-- --	-- --	80 90	-- --	-- --	-- --	-- --
0.0500 to 0.0599	-- --	-- 100	-- 160	-- --	160 --	-- --	-- --	-- --	-- --
0.0600 to 0.0699	150 153	-- --	-- 170	-- 250	-- --	-- 367	-- --	-- --	-- --
0.0700 to 0.0799	-- --	-- 100	-- 160	-- --	-- --	-- --	71 --	-- --	-- --
0.0800 to 0.0899	--	--	--	280	--	400	--	--	--
0.0900 to 0.0999	--	--	--	--	--	--	--	--	--
0.1000 to 0.1099	--	--	--	--	--	--	110	--	--
0.1100 to 0.1199	270	--	--	--	--	287	--	--	--
0.1200 to 0.1299	--	145	--	--	--	445	138	--	--
0.1300 to 0.1399	--	--	250	--	--	326	--	--	--
0.1400 to 0.1499	--	--	--	--	--	--	--	--	--
>0.1500	450	--	--	--	--	--	--	--	--

TABLE VIII. - SUMMARY OF FILM WEIGHT LOSS RATES AT 315° C AND 350° C FOR THE NINE DIFFERENT TYPES OF POLYIMIDES EVALUATED.

Type of polyimide	Rate of film weight loss, percent/hr	
	Exposure temperature	
	315° C	350° C
PIC-1	0.053 ± 0.014	.26 ± 0.03
PIC-2	.042 ± .011	.20 ± .07
PIC-3	.042 ± .017	.20 ± .10
PIC-4	.087 ± .015	34 ± .03
PIC-5	.043 ± 0.14	.27 ± .08
PIC-6	.079 ± .030	.40 ± .15
PIC-7	.025 ± .009	.13 ± .04
PMR-15	.175*	-----
PMR-II	.036*	-----

*One test only.

TABLE IX. - EXPOSURE TIMES AT 315° C WHEN CRACKING AND SPALLING OF POLYIMIDE FILMS WAS OBSERVED TO OCCUR

Type of polyimide	Weight of film, g	Total exposure time, hr	Time when cracks observed, hr	Time when spalling observed, hr
PIC-1	0.1272	523	214	447
	.0946	447	334	447
	.0640	523	523	none
	.0400	523	523	none
	.0337	523	none	none
PIC-2	0.0900	766	none	none
	.0884	766		
	.0834	766		
	.0782	530		
	.0729	766		
	.0400	501		
	.0361	523		
	.0300	523		
	.0294	523		
PIC-3	0.0700	501	none	none
	.0640	523		
	.0528	530		
	.0499	506		
	.0364	523		
PIC-4	0.1545	170	100	170
	.0862	526	214	334
	.0315	523	115	334
	.0207	523	none	none
PIC-5	0.0767	523	none	none
	.0696	530		
	.0534	523		
	.0503	501		
	.0397	530		
PIC-6	0.1416	144	144	none
	.1132	311	311	311
	.0990	214	115	214
	.0940	47	47	47
	.0789	214	115	214
	.0276	523	214	334
PIC-7	0.1211	530	none	none
	.0818	501		
	.0711	523		
	.0557	523		
	.0521	523		
	.0507	523		
	.0366	530		
PMR-15	0.1500	313	168	216
PMR-II	0.1500	313	none	none

TABLE X. - EXPOSURE TIMES AT 350° C WHEN CRACKING AND SPALLING OF POLYIMIDE FILMS WAS OBSERVED TO OCCUR

Type of polyimide	Weight of film, g	Total exposure time, hr	Time when cracks observed, hr	Time when spalling observed, hr
PIC-1	0.1946	94	94	94
	.1122	146	146	146
	.0529	184	184	184
	.0515	213	213	213
PIC-2	0.1237	184	none	none
	.0700	184	none	
	.0510	184	none	
	.0421	213	146	
	.0374	213	213	
	.0330	100	none	
PIC-3	0.1338	94	47	94
	.0700	184	none	none
	.0645	184	none	none
	.0533	146	97	146
PIC-4	0.0868	94	47	94
	.0663	94	47	94
PIC-5	0.0568	184	184	none
	.0455	213	none	
	.0434	213		
	.0407	100		
	.0361	184		
	.0338	184		
PIC-6	0.1301	48	24	48
	.1235	48	24	24
	.1186	48	24	48
	.0800	20	20	20
	.0665	70	46	70
PIC-7	0.1200	213	213	none
	.1000	94	47	94
	.0764	184	47	94
	.0346	213	none	none

TABLE XI. - CLASSIFICATION OF THE NINE DIFFERENT TYPES OF POLYIMIDES INTO WEIGHTLOSS AND ADHERENCE EQUIVALENCY GROUPS FOR FILMS EXPOSED TO 315° C or 350° C FOR LONG TIME DURATIONS.

Polyimide type	Weight loss rate	Adherence
	Group classification *	
PIC-1	3	3
PIC-2	2	1
PIC-3	2	2
PIC-4	4	3
PIC-5	2	1
PIC-6	4	3
PIC-7	1	2
PMR-15	5	3
PMR-II	2	2

*The higher the group number, the worse the results.

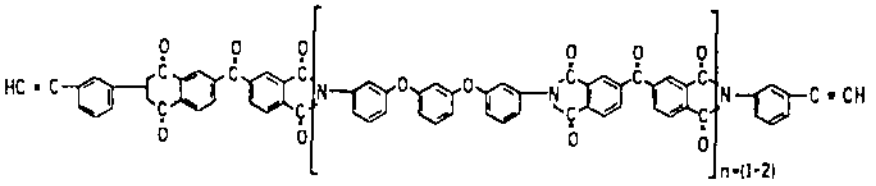


Figure 1. - Structure of polyimide PIC-6.

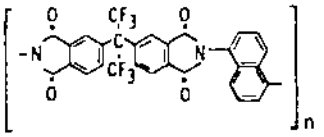


Figure 2. - Structure of polyimide PIC-7.

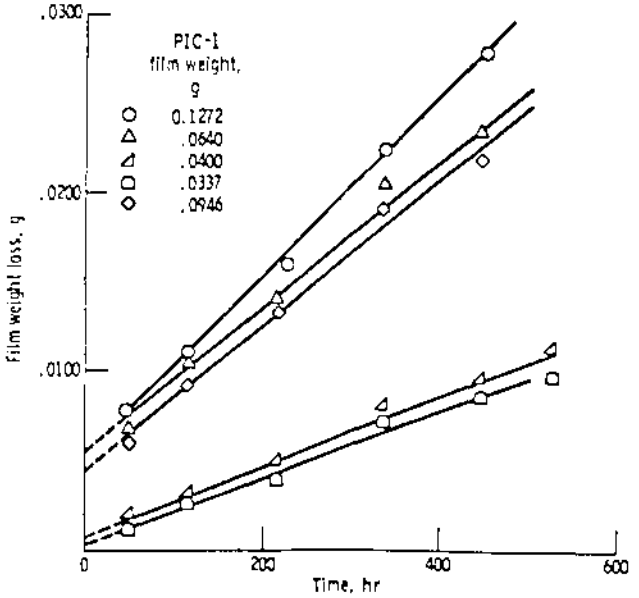


Figure 3. - Polyimide film type PIC-1 weight loss at 315° C as a function of exposure time for five different film weights.

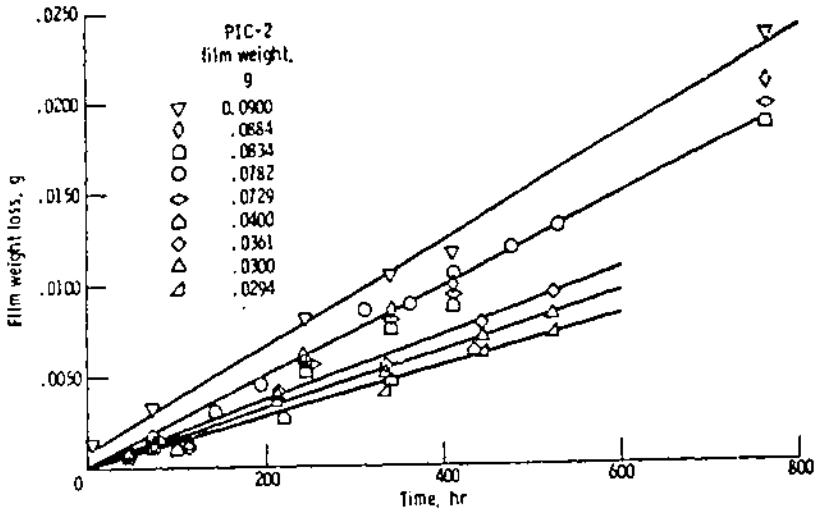


Figure 4. - Polyimide film type PIC-2 weight loss at 315° C as a function of exposure time for nine different film weights.

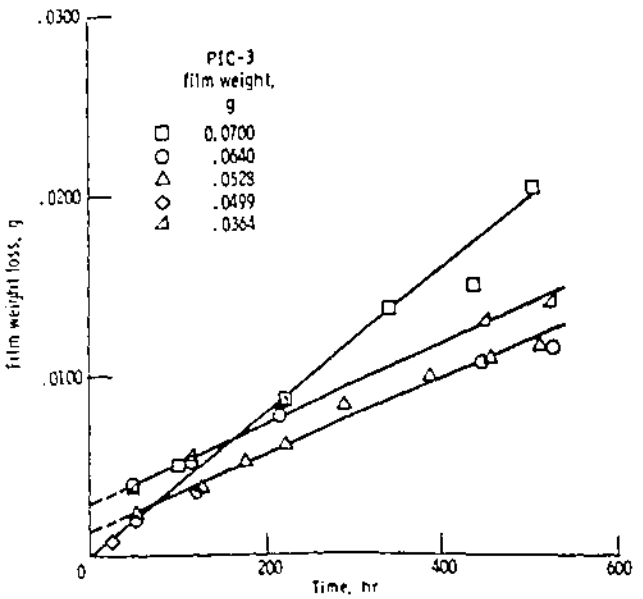


Figure 5. - Polyimide film type PIC-3 weight loss at 315° C as a function of exposure time for five different film weights.

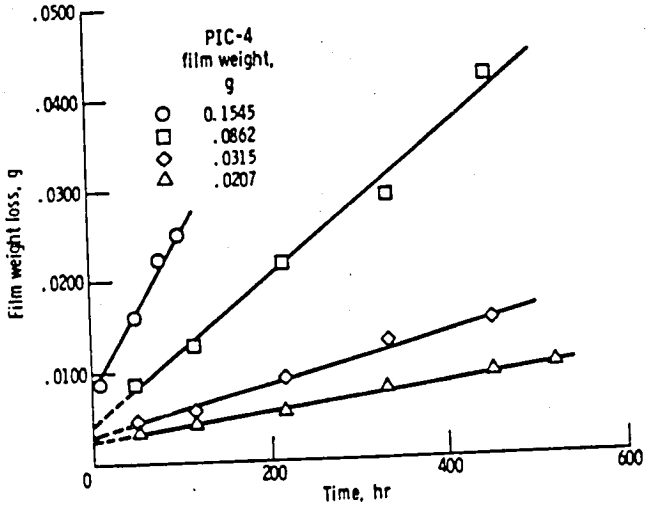


Figure 6. - Polyimide film type PIC-4 weight loss at 315° C as a function of exposure time for four different film weights.

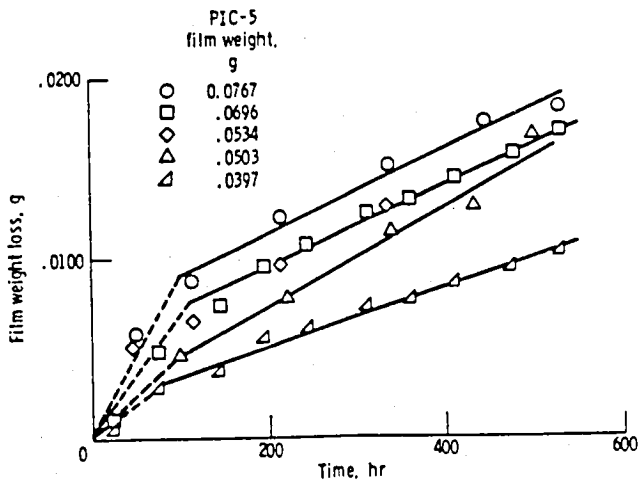


Figure 7. - Polyimide film type PIC-5 weight loss at 315° C as a function of exposure time for five different film weights.

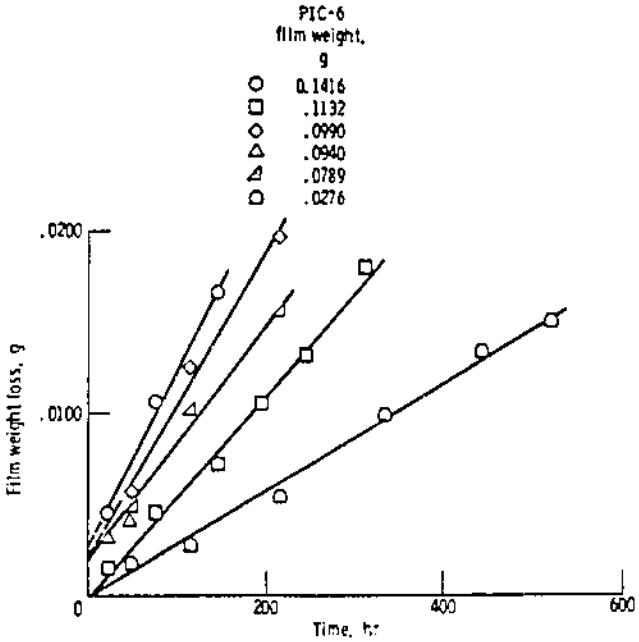


Figure 8. - Polyimide film type PIC-6 weight loss at 315° C as a function of exposure time for six different film weights.

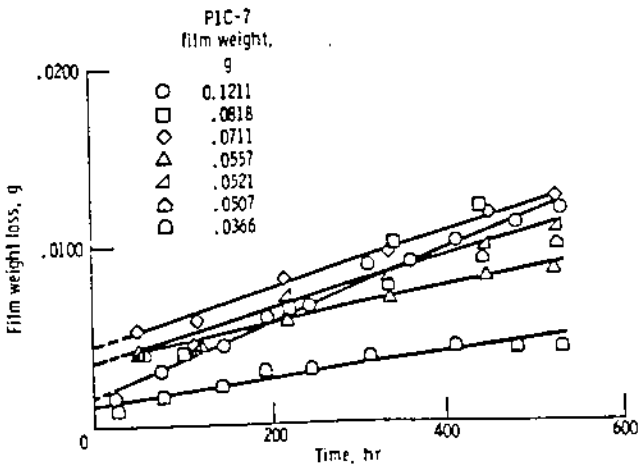


Figure 9. - Polyimide film type PIC-7 weight loss at 315° C as a function of exposure time for seven different film weights.

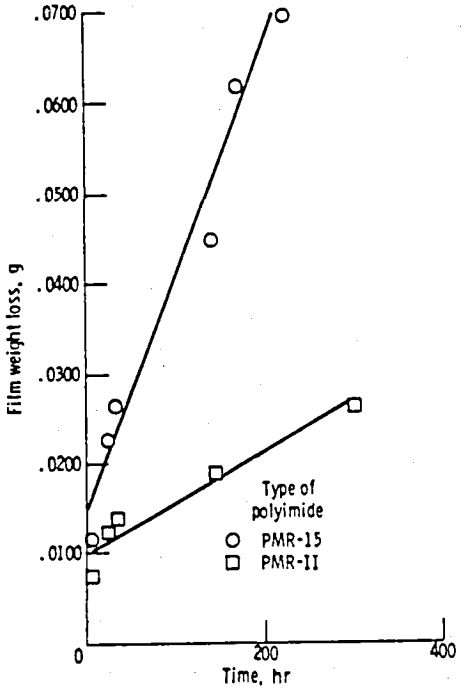


Figure 10. - Polyimide film weight loss at 315° C as a function of exposure time for polyimide types PMR-15 and PMR-II.

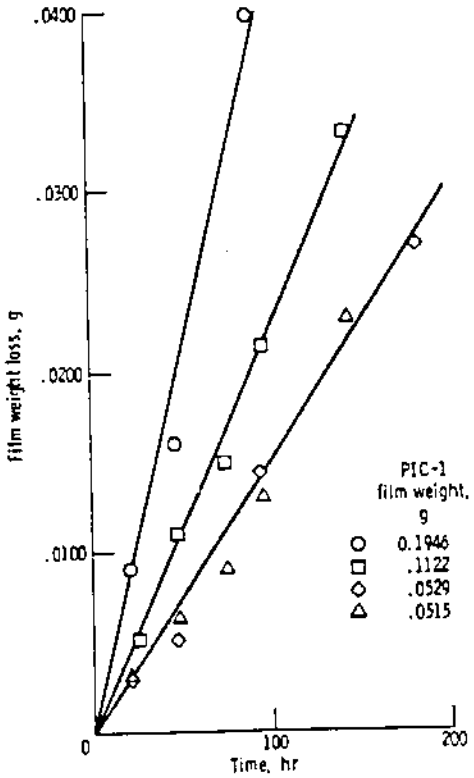


Figure 11. - Polyimide film type PIC-1 weight loss at 350° C as a function of exposure time for four different film weights.

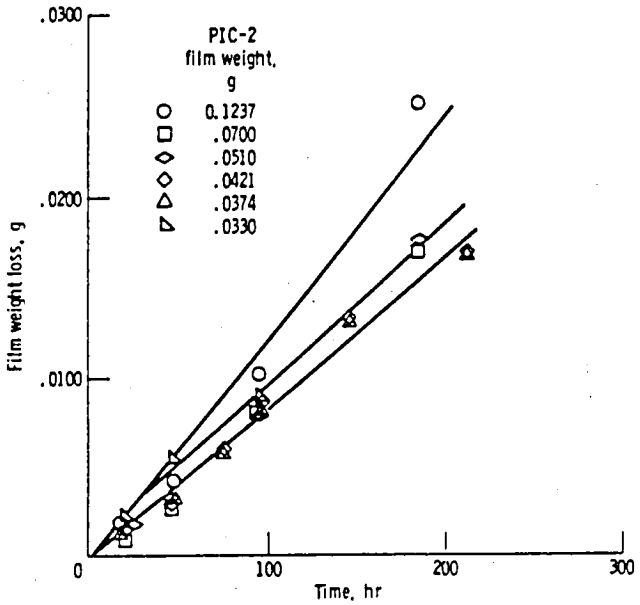


Figure 12. - Polyimide film type PIC-2 weight loss at 350° C as a function of exposure time for six different film weights.

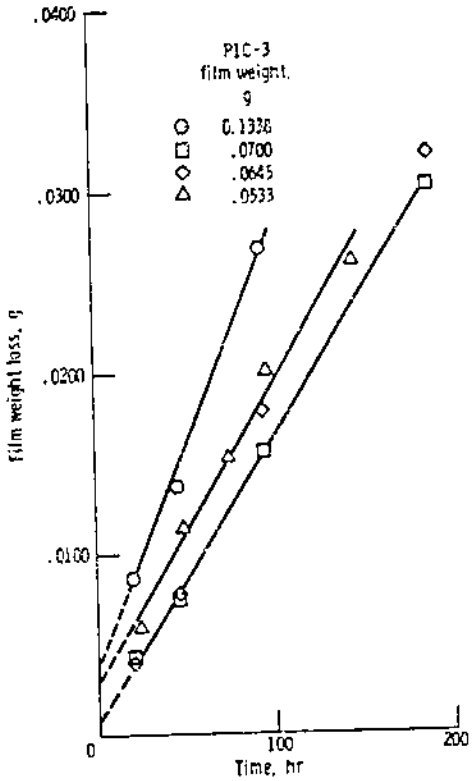


Figure 13. - Polyimide film type PIC-3 weight loss at 350° C as a function of exposure time for four different film weights.

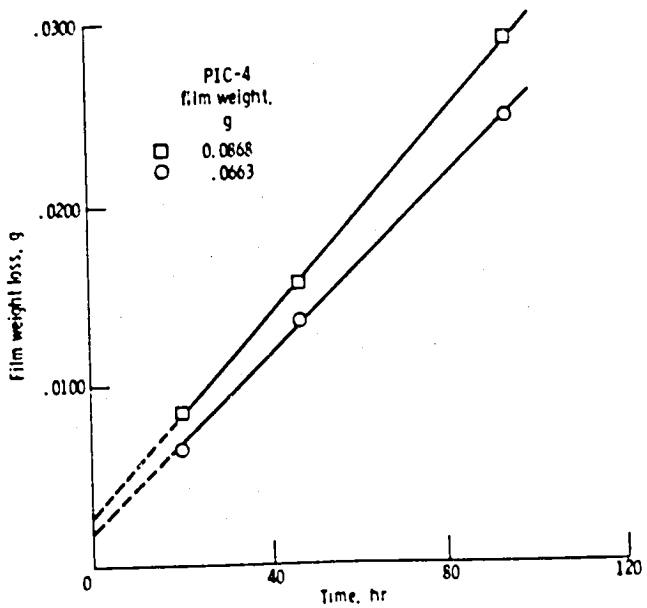


Figure 14. - Polyimide film type PIC-4 weight loss at 350° C as a function of exposure time for two different film weights.

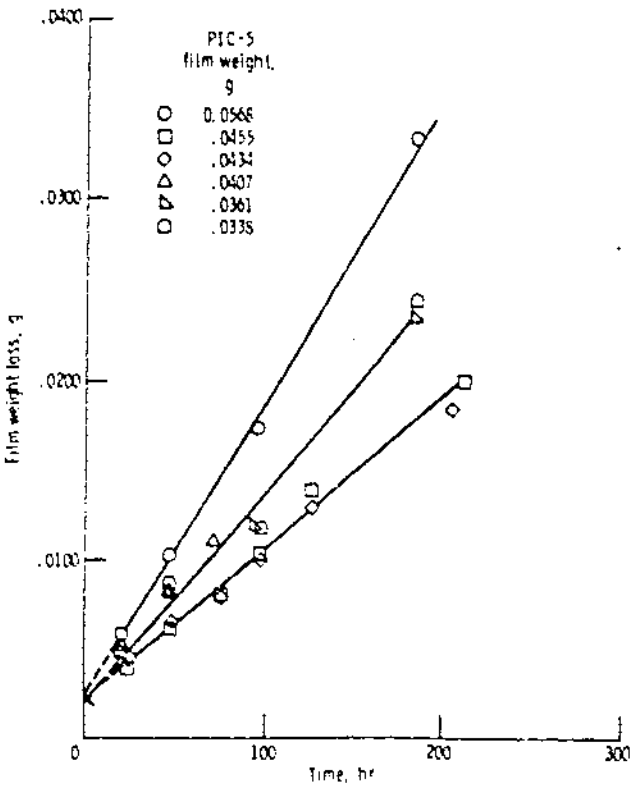


Figure 15. - Polyimide film type PIC-5 weight loss at 350° C as a function of exposure time for six different film weights.

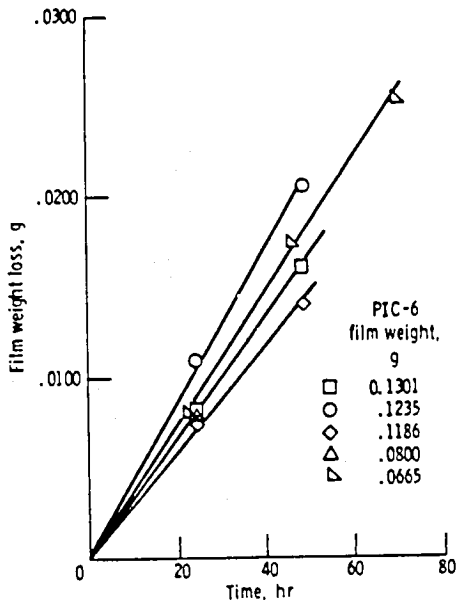


Figure 16. - Polyimide film type PIC-6 weight loss at 350° C as a function of exposure time for five different film weights.

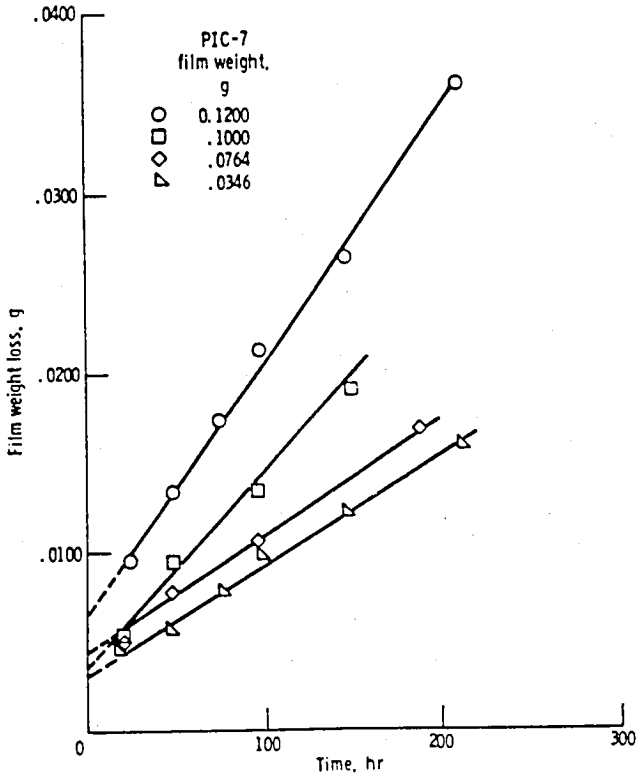


Figure 17. - Polyimide film type PIC-7 weight loss at 350° C as a function of exposure time for four different film weights.

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