

THERMOCHEMICAL CHARACTERIZATION OF SOME THERMALLY
STABLE THERMOPLASTIC AND THERMOSET POLYMERS

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INTRODUCTION

The purpose of this study was to assess the relative thermal stability, flammability, and other related thermochemical properties of some typical state-of-the-art and candidate experimental thermoset and thermoplastic materials and to assess their potential use as facesheet for panels, moldings, thermoformed parts, and decorative films in aircraft interiors. State-of-the-art materials that were evaluated included: acrylonitrile butadiene styrene (ABS), and epoxy.

Advanced thermoplastic materials evaluated included: 9,9-bis(4-hydroxyphenyl)-fluorene polycarbonate-poly(dimethylsiloxane) block polymer (BPFC-DMS), polyethersulfone (PES), polyphenylene sulfide (PPS), and polyaryl sulfone (PAS).

Advanced thermoset materials evaluated included modified phenolics, bismaleimide, and polyaromatic melamine resins.

Typical applications for the thermoplastics materials within a wide body aircraft are moldings for passenger service units, luminaries, trays, shrouds, and other decorative or functional parts. Applications for the thermoset materials include the facesheets for the interior composite panels and partition walls and are shown in Figures 1 and 2. Aircraft interior materials must meet current regulatory requirements [1]. Both the state-of-the-art and advanced materials are being studied by industry and government agencies [2-14] in order to identify materials that would offer improved fire resistance and, upon combustion, produce less smoke, irritating fumes, and toxic gases. The objectives of this study and the flammability parameters evaluated are shown in Table 1.

SYMBOLS

The International System of Units (SI) is used.

D _s	specific optical density, $D_s = \frac{(131.58) \text{ Log}_{10} 100}{T}$
D _m	specific optical density, maximum
LOI	limiting oxygen index, $O_2 / (O_2 + N_2)$
TGA	thermogravimetric analysis
Y _c	char yield, percent weight remaining
T _g	glass transition temperature
T	percent light transmittance = $\frac{100}{\text{antilog} \left(\frac{D_s}{132} \right)} = \frac{100}{10^{(D_s/132)}}$
T _m	melt temperature

EXPERIMENTAL

Thermoplastic Polymers

The thermoplastic polymers evaluated included both state-of-the-art and other high temperature polymers. The chemical structure, glass transition temperature (T_g) and melt temperature (T_m) of the polymers evaluated are shown in Table 2. Polymers were evaluated as injection molded or extruded sheet. The detailed chemistry of the polymers evaluated has been described previously in the following references: acrylonitrile butadiene styrene (state-of-the-art) [15]; 9,9-bis(4-hydroxyphenyl)-fluorene polycarbonate-poly(dimethylsiloxane) block polymer [16-19]; polyether sulfone [20]; polyphenylene sulfide [21]; and polyaryl sulfone [22].

Thermomechanical properties - The glass transition (T_g) and melt (T_m) temperatures were determined by the DuPont thermal mechanical analyzer (TMA), Model 941, with the DuPont thermal analyzer, Model 900. Measurements were made in the penetration or compression mode, using a hemispherical tip probe.

In addition to studies conducted on the TMA, differential scanning calorimeter (DSC) studies were conducted with a DuPont Model 900 DSC on polyphenylene sulfide, polyaryl sulfone, and polyether sulfone. In the DSC for polyether sulfone and polyaryl sulfone, an endotherm was observed when the temperature of thermal decomposition was attained.

The thermal properties of polyphenylene sulfide are shown in Figures 3 and 4. Two endotherms and one exotherm were observed. The DSC thermogram (Figure 3) shows an endotherm (a) at 90°C . This temperature corresponds to the glass transition temperature ($T_g = 89^\circ\text{C}$) determined by TMA as shown in Figure 4. However, an enthalpy change is not associated with T_g as it is not a first-order transition. Thus, the transition at 90°C must be due to melting, which is a first-order transition. The data indicate that polyphenylene sulfide is highly crystalline. Previous studies [23] have shown a glass transition temperature of 85°C . The second endotherm, Figure 3 (c) at 271°C , may be due to some thermal decomposition, even though this weight loss was not evident in the TGA thermogram because of the rapid heating rate ($40^\circ\text{C}/\text{min}$). The exotherm, Figure 3 (b) at 119°C , is due to crosslinking of polyphenylene sulfide. According to previous studies [23], crosslinking with recrystallization occurs in the presence of air. Polyphenylene sulfide again forms a glass on crosslinking.

In summary, polyphenylene sulfide is thermally very stable. It undergoes interesting and unusual transitions in physical state when heated, which accounts for its good high-temperature mechanical properties. The extruded pellets of polyphenylene sulfide are highly crystalline, with a melting point of 89°C . Crosslinking with the formation of an amorphous glass occurs at 119°C . The glass temperature of the second glass region is 250° to 271°C .

Thermogravimetric Analyses - Thermal analyses of the polymers were conducted on a DuPont 950 thermogravimetric analyzer (TGA) using nitrogen atmospheres with a sample size of 10 mg. The thermogravimetric analysis data of 40°C/min heating rate in nitrogen is shown in Figure 5.

9,9-bis(4-hydroxyphenyl)-fluorene polycarbonate-poly(dimethylsiloxane) block polymer is the most stable transparent thermoplastic polymer and gives the highest char yield. Polyphenylene sulfide is the most stable polymer tested. According to previous studies [23], thermooxidative degradation of polyphenylene sulfide occurs rapidly above 520°C. This is in excellent agreement with the present studies, during which degradation occurred at approximately 500°C.

Processing Studies - Processing studies were conducted to determine the feasibility of processing the advanced polymers. The purpose was to evaluate processing parameters for molding and to optimize the parameters for molding specimens with optimum thermophysical properties. Processing studies were conducted on 9,9-bis(4-hydroxyphenyl)-fluorene polycarbonate-poly(dimethylsiloxane), polyether sulfone, polyphenylene sulfide, and polyaryl sulfone. The typical molding parameters are summarized in Table 3.

9,9-bis(4-hydroxyphenyl)-fluorene polycarbonate-poly(dimethylsiloxane) temperature profile was set at the rear nozzle at 298°C, in the middle nozzle at 321°C, and at the front nozzle at 315°C. The mold temperature was 121°C; the mold surface temperature was 107°C. The injection pressure was 89635 kN/m², the injection speed 1 sec, the injection pressure hold-time 10 sec, the feed time 25 sec, the screw rpm was 160, and the pressure in the runner feeding disk was 7998 kN/m². Disks with good optical clarity were produced using the above molding procedure.

Molding of the other materials (polyether sulfone, polyphenylene sulfide, and polyaryl sulfone) was accomplished in a conventionally heated molding press using conventional compression molding techniques. All of the specimens were made in a steel mold consisting of a ring segment 1.905 cm long, 7.620 cm i.d., and 1.270 cm wall, screwed to a 0.953 cm flat plate. The ram slip fitted into the ring.

Molding tests were made of polyether sulfone and polyphenylene sulfide to establish a workable temperature and pressure cycle that would form flat panels in a thickness range of 0.254 to 0.635 cm. All materials were in the form of pellets of about 0.381 cm long by 0.254 cm diam., and each had a melting point of approximately 260°C. Materials were kept in a 148°C oven and removed just prior to molding.

Polyether sulfone appears to have satisfactory molding characteristics for producing sheet stock. Initial trials were not successful because the pellets were not dried. Once the 148°C drying process was employed, satisfactory specimens could

be provided over a reasonable range of temperature and pressure conditions, as shown in Table 3. Polyphenylene sulfide was slightly more difficult to mold into satisfactory specimens than polyether sulfone. The polyphenylene sulfide has a somewhat higher melting point than polyether sulfone (approximately 267°C). However, it becomes liquid at the melting temperature, and even low pressures force it out of the mold completely. Yet, at slightly lower temperatures, the material does not consolidate completely under very high pressures, i.e., 27.58 MN/m². Optimum molding conditions and satisfactorily molded specimens were obtained with a molding pressure of 6.8 MN/m² and molding temperature of 238° to 260°C. Polyaryl sulfone was molded satisfactorily at a molding temperature of 340° to 370°C and molding pressure of 7.5 MN/m².

Physical Properties - Some of the physical properties of the polymers are shown in Table 4. Properties that were evaluated included specific gravity, tensile strength, ultimate elongation, heat deflection temperature, flexural strength, flexural modulus, impact strength, and compressive strength. The physical properties of the advanced polymers are within the range of the desired properties of thermoplastic materials for aircraft interior components [24].

Oxygen Index - The oxygen index of the polymers was determined per ASTM D-2863 and is shown in Table 5. Polyphenylene sulfide and 9,9-bis(4-hydroxyphenyl)-fluorene polycarbonate-poly(dimethylsiloxane) block polymer had the highest oxygen index of the thermoplastic polymers tested.

Smoke Evolution - Smoke evolution from the polymers was determined using the NBS-Aminco smoke density chamber. The procedure and test method used were essentially those described by NFPA-258-T [25]. A detailed description of the NBS smoke chamber can be found in Reference 26. The D_s values are obtained from individual test data and then averaged. The test results obtained are presented in Figure 6. Tests were conducted at a heat flux of 2.5 W/cm² under flaming conditions.

Polyether sulfone exhibited extremely low smoke evolution. The other advanced polymers also exhibited significantly lower smoke evolution than the state-of-the-art acrylonitrile butadiene styrene. In accordance with proposed regulations [3] smoke evolution for aircraft interior materials should not exceed D_s 100 in 4 minutes.

Effect of Char Yield on Oxygen Index - Previous studies [27,28] have shown a correlation between the flammability properties of some polymers and their char yield. Figure 7 compares the oxygen index of the polymers evaluated with their relative anaerobic char yields. It can be seen that, in general, polymers with high char yield exhibit a high oxygen index. Polyphenylene sulfide exhibited the highest oxygen index and correspondingly had the highest char yield of the polymers evaluated. The test specimen for the oxygen index

test was 0.25 cm thick - 5.0 cm wide - 12.0 cm high. The calculated or theoretical oxygen index indicated by from reference 28.

Thermoset Polymers

The thermoset polymers evaluated included both state-of-the-art (epoxy) and other high temperature resins. The chemical structure and glass transition temperature for these resins are shown in Table 6. The resins were evaluated both as neat resins and preimpregnated with glass cloth. The epoxy resin is utilized as the resin for the facesheets of typical state-of-the-art aircraft interior composite panels. The epoxy resin is a diglycidyl ether of bisphenol A cured with methylene dianiline.

The other resins are currently being evaluated as a possible replacement to the epoxy resin utilized.

The phenolic resin evaluated was a modified phenolic resin commercially available.

The bismaleimide resin evaluated was also commercially available. Its chemistry has been described previously [29].

The chemistry of the polyaromatic melamine resin evaluated has been described previously [30].

Processing Studies - The resins were utilized in the fabrication of prepreg facesheets which are used in the construction of both state-of-the-art and experimental aircraft interior panels. The fabrication and properties of composite panels have been described previously [31].

Test specimens were prepared as single plies of resin coated on 181 glass cloth. The glass was coated with resin and processed as follows:

Epoxy - The glass cloth is impregnated with the resin. The "pre-preg" is placed in a press and is cured for 12 minutes at 160°C and 689 kN/m². This is followed by molding in a vacuum bag at 126°C for one hour and 50 torr minimum vacuum bag pressure. The cured skin contains approximately 47% by weight resin.

Phenolic - Processing was identical to the epoxy described above.

Bismaleimide - The glass cloth is impregnated with the resin and is cured in a press for one hour at 182°C and 689 kN/m² pressure. After removal from the press, the prepreg is postcured in an oven for 16 hours at 250°C.

Polyaromatic melamine - The glass cloth is impregnated with the resin and is cured in a press for one hour at 204°C and 1379 kN/m² pressure. After cooling in the press to room temperature, it is postcured in an oven for 16 hours at 225°C.

Both the bismaleimide and polyaromatic melamine resins require longer processing times and longer postcure times than the phenolics or epoxy resins to remove either volatile compounds from condensation reactions or high boiling solvents

like N-methyl pyrrolidone. However, these resins have inherent higher fire-resistant properties as shown by the higher oxygen index of the glass fabric/resin laminates.

Thermochemical characterization - The resins and the cured glass fabric/resin laminates were subjected to the following thermochemical and flammability studies in order to assess their relative fire-resistance: (1) thermogravimetric analysis of the resin, (2) oxygen index of the composite, and (3) smoke evolution of the composite.

The relative thermal stability of the resins in an anaerobic environment is shown in Figure 8. The polyaromatic melamine had the highest char yield of the polymers tested.

The smoke evolution from the glass fabric/resin laminates was determined using similar procedure as outlined previously. Both the phenolic and bismaleimide resins exhibited very low smoke evolution.

The same relationship of anaerobic char yield and oxygen index was shown in the thermoset polymers as in the thermoplastic polymers as indicated in Figure 9. The char yield values indicated are of the neat resin and the glass fabric/resin laminate. The correlation is not as evident as in the thermoplastic polymers since the oxygen index on these resins was determined on single-ply specimens of glass fabric/resin.

CONCLUSIONS

Material selection requires the exercise of engineering judgment based on the data available or obtained by adequate testing and the potential consequences of any fire which may occur in an enclosure such as a wide body jet. One must be fully cognizant of all aspects of the material and carefully weigh all trade-offs relating to cost, weight, maintainability, physical properties and flammability, including toxicity, smoke, and fuel contribution.

The selected material must exhibit the following properties when subjected to the combination of flammability tests, some of which are discussed previously:

- high ignition temperature
- ease of extinguishability
- ability to absorb high radiant energy
- ability to contain flame progression (flame travel rate)
- low rate of heat release/fuel contribution
- low or non-combustibility (lack of violent reaction to proximity of heat or ignition source)
- smoke emission
 - high evolution temperature
 - low percent obscuration and density
- toxicity emission
 - high evolution temperature
 - minimum toxic gases released
 - low concentration

In this study some of the processability and flammability parameters of the polymers were determined.

To rank the materials one should consider all of the materials parameters and assign weight to each specific parameter or measurement.

Based on the flammability data alone and assuming equal weight assignment to each flammability parameter (LOI, Yc, and T) one can calculate each measurable parameter in terms of percent (100% having the highest fire-resistant properties). Assuming this equal weight assignment, one could rate the polymers in order of increased fire safety as shown in Table 7: thermoplastics - acrylonitrile butadiene styrene, polyaryl sulfone, polyether sulfone, 9,9-bis(4-hydroxyphenyl)-fluorene polycarbonate-poly(dimethylsiloxane), and polyphenylene sulfide; thermosets - epoxy, polyaromatic melamine, phenolic, and bismaleimide.

A correlation was established between the oxygen indices of the polymers and their anaerobic char yields. Generally, polymers with high anaerobic char yield exhibited high oxygen index. There was no relationship noted between the thermo-mechanical (Tg, Tm) properties of the polymers and their flammability characteristics.

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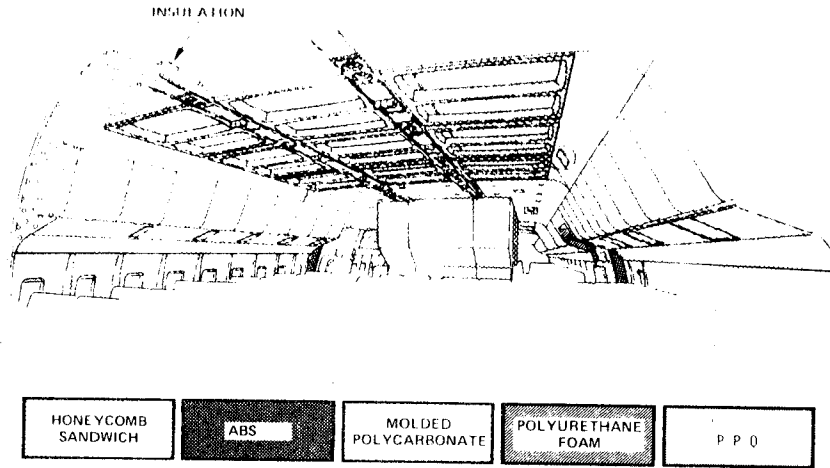


Fig. 1.- Passenger cabin.

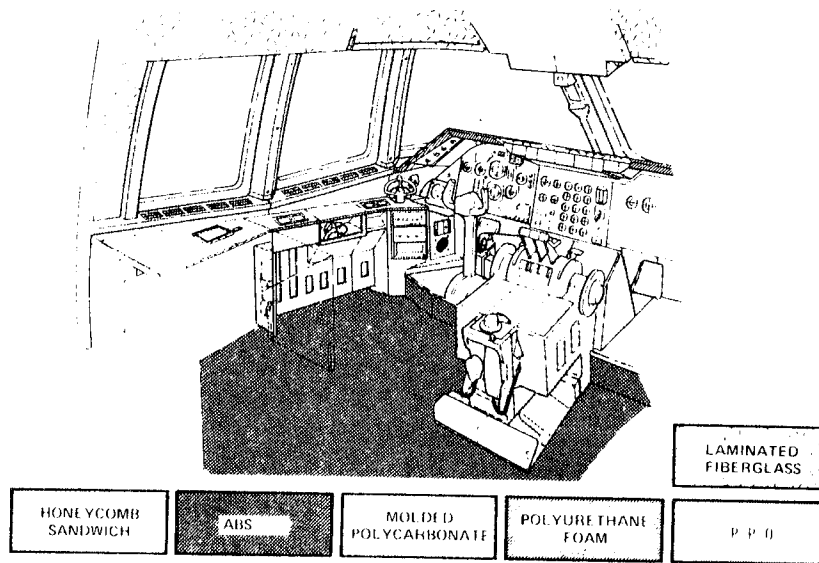


Fig. 2.- Flight station.

(HEATING RATE = 10°C/min., AIR)
TEMPERATURES (°C) CORRECTED TO THE MELTING POINT OF INDIUM.

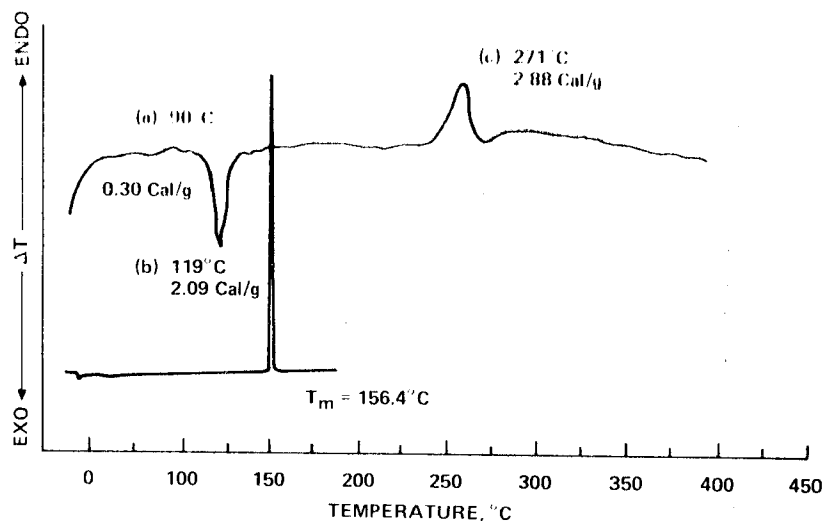


Fig. 3.- Thermal properties of polyphenylene sulfide (differential scanning calorimeter).

(THERMAL MECHANICAL ANALYSIS-PENETRATION)
(HEATING RATE = 15°C/min., AIR)

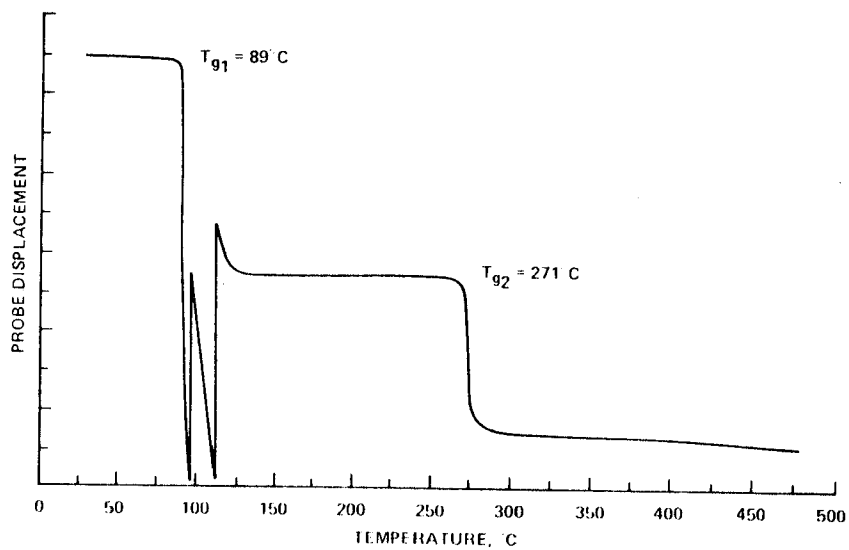


Fig. 4.- Thermal properties of polyphenylene sulfide (thermomechanical analysis-penetration).

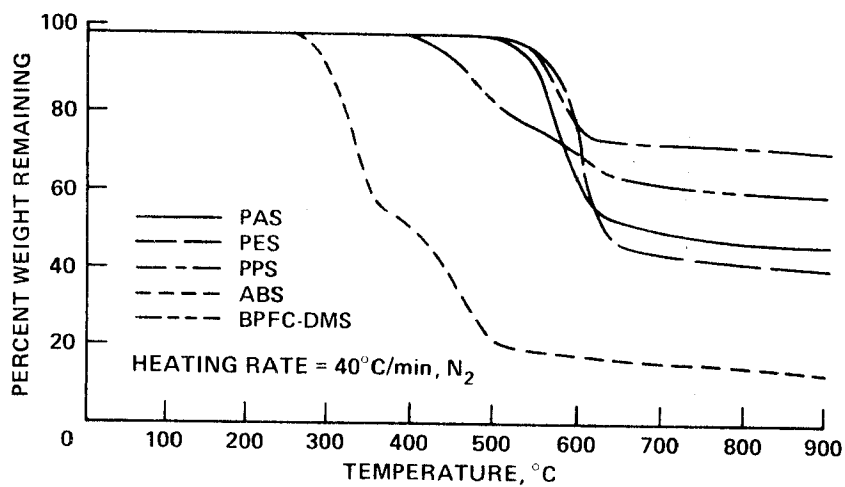


Fig. 5.- Dynamic thermograph for thermoplastics.

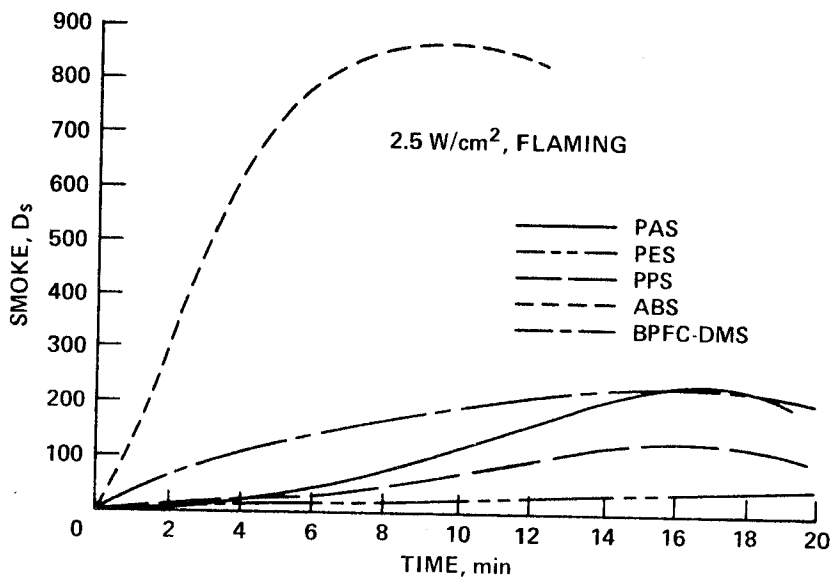


Fig. 6.- Smoke concentration histories for thermoplastics.

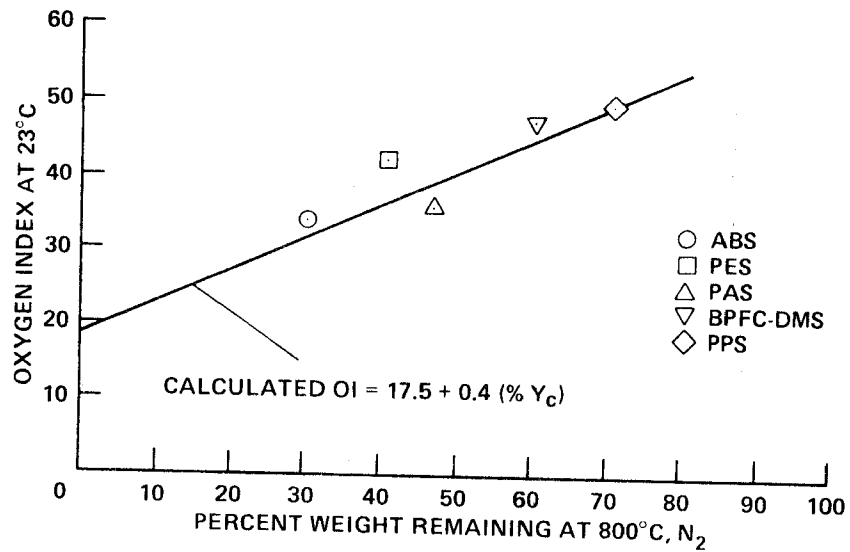


Fig. 7.- Effect of char yield of thermoplastic polymers on oxygen index.

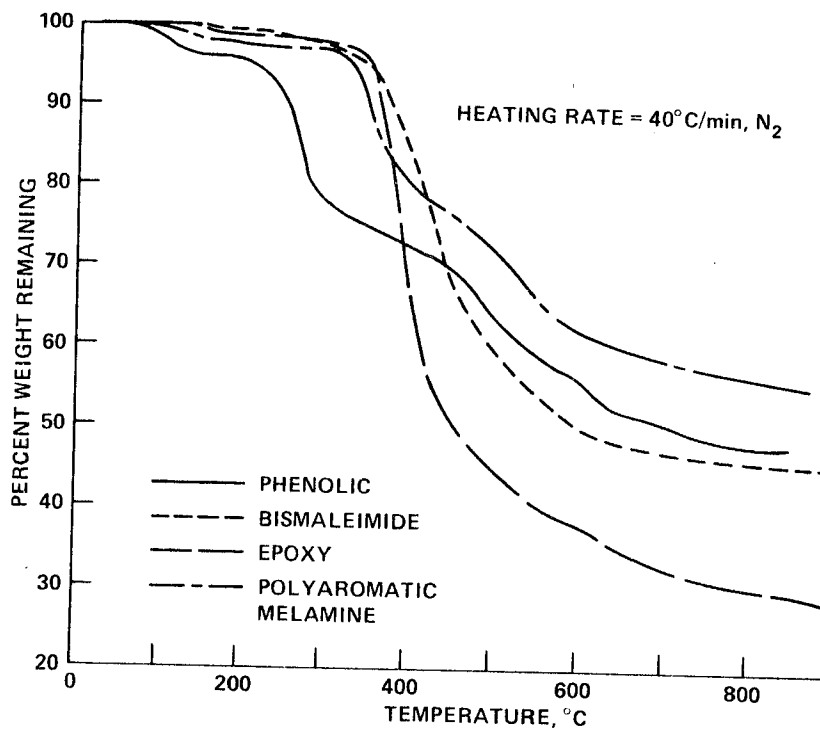


Fig. 8.- Dynamic thermograph for thermosets.

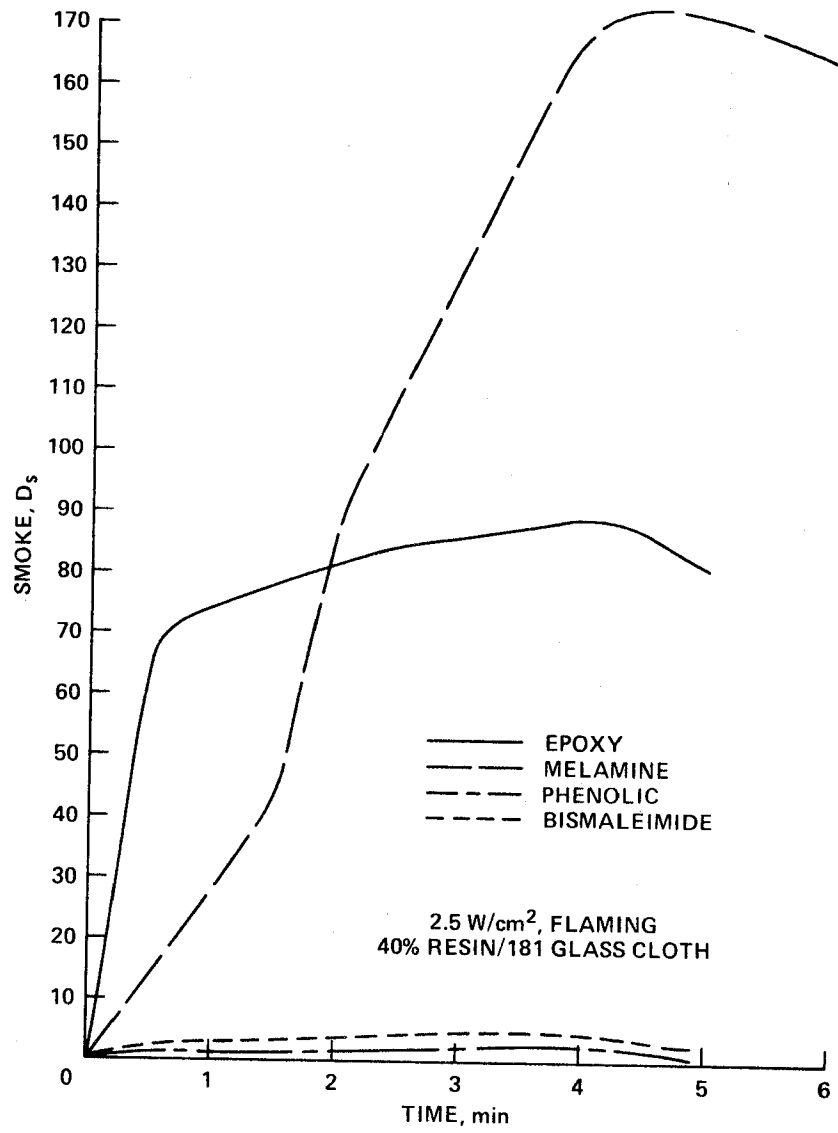


Fig. 9.- Smoke concentration histories for thermoset resins.

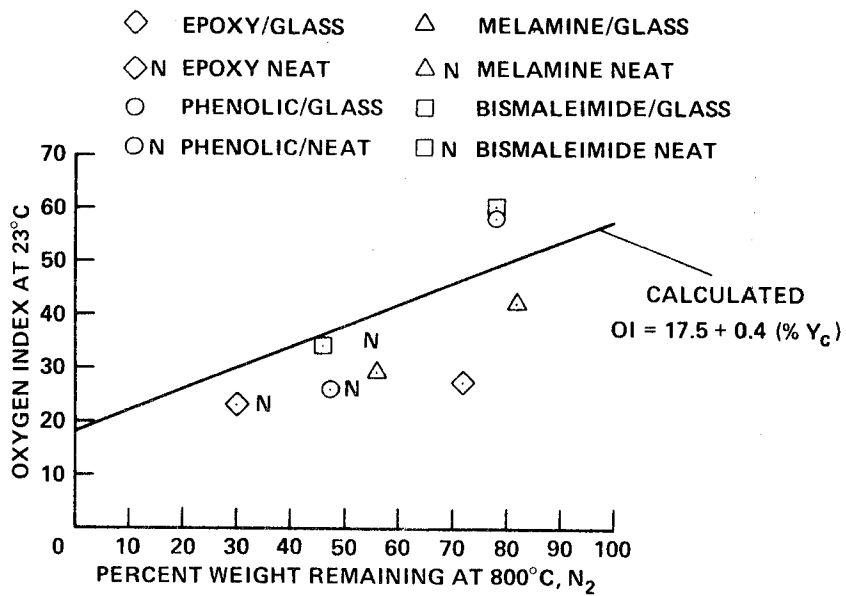


Fig. 10.- Effect of char yield of thermosetting polymers on oxygen index.

OBJECTIVE

DETERMINE THERMAL-CHEMICAL AND FLAMMABILITY PROPERTIES OF TYPICAL STATE-OF-THE-ART AND OTHER ADVANCED THERMOPLASTICS AND THERMOSETS IN ORDER TO ASSESS THEIR RELATIVE FIRE RESISTANCE

SCOPE

- DETERMINE PROPERTIES
- THERMOMECHANICAL
 - GLASS TRANSITION TEMPERATURE
 - MELT TEMPERATURE
 - CHAR YIELD
- PROCESSING
 - MOLDING PARAMETERS
- FLAMMABILITY
 - OXYGEN INDEX
 - SMOKE EVOLUTION
- PHYSICAL MECHANICAL
 - TENSILE
 - COMPRESSION
 - ELONGATION

Table 1 Program Objectives

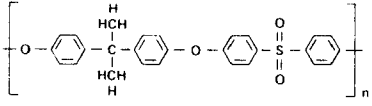
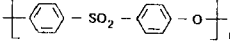
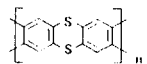
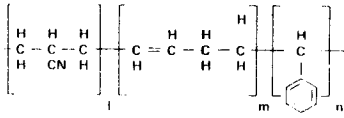
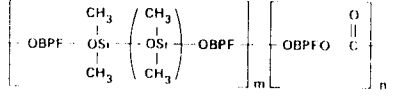
POLYMER	POLYMER STRUCTURE	T _g C	T _m C
POLYARYL SULFONE (PAS)		283	310
POLYETHER SULFONE (PES)		220	228
POLYPHENYLENE SULFIDE (PPS)		87	276
ACRYLONITRILE BUTADIENE STYRENE (ABS)		80	
9,9 BIS (4 HYDROXY PHENYL) FLUORENE POLYCARBONATE POLY (DIMETHYL SILOXANE) BLOCK POLYMER (BPFC DMS)		215	360 370

Table 2 Typical Structure of Thermoplastic Polymers

MOLDING PARAMETER	PES	PPS	PAS	BPFC DMS
MOLDING CYCLE:				RECIPROCATING SCREW
STABILIZE PRESSURE (minutes)				NOZZLE TEMP. (°C)
RANGE	3-5	3-8	2-10	FRONT: 315
OPTIMUM	4	5	4	MIDDLE: 321
MAINTAIN PRESSURE (minutes)				REAR: 298
RANGE	5-7	4-10	4-30	
OPTIMUM	5	5	6	
MOLDING PRESSURE (MN/m ²)				
RANGE	3.4-27.5	3.4-27.5	1.9-58.5	7.5-8.5
OPTIMUM	6.8	6.8	7.5	7.9
MOLDING TEMPERATURE (°C)				MOLD TEMP. (°C)
INITIAL,				
RANGE	232-316	260-289	340-370	120
OPTIMUM	274	277	347	
FINAL,				
RANGE	238-307	260-288	315-370	
OPTIMUM	260	273	327	
LOWEST DURING CYCLE,				
RANGE	213-310	238-279	310-330	
OPTIMUM	252	271	315	

Table 3 Typical Molding Parameters for Thermoplastics

POLYMER	PROPERTIES							
	SPECIFIC GRAVITY ASTM D792 (g/cm ³)	TENSILE STRENGTH @ 23°C ASTM D1708 (MN/m ²)	ULTIMATE ELONGATION @ 23°C ASTM D1708 (%)	HEAT DEFLECTION TEMPERATURE @ 182 KN/m ² ASTM D648 (°C)	FLEXURAL STRENGTH @ 23°C ASTM D790 (MN/m ²)	FLEXURAL MODULUS @ 23°C ASTM D790 (GN/m ²)	IMPACT STRENGTH NOTCHED (kJ/m ²) @ 23°C ASTM D256	COMPRESSIVE STRENGTH @ 23°C ASTM D1621-64 (GN/m ²)
POLYARYL SULFONE (PAS)	1.25	68.95	40	166.0	103.43	2.34	69.4	93.08
POLYETHER SULFONE (PES)	1.26	75.85	70	199.0	110.32	2.41	85.4	82.74
POLYPHENYLENE SULFIDE (PPS)	1.36	74.47	3	136.7		4.4	16.2	---
ACRYLONITRILE BUTADIENE STYRENE (ABS)		37.92	2	110.0		1.72-3.10	80.1	
9,9-BIS(4-HYDROXYPHENYL) FLUORENE POLYCARBONATE POLY (DIMETHYLSILOXANE) BLOCK POLYMER (BPFC DMS)	1.17	46.89-51.23	20-48	200-210	12.89-14.76	1.66-2.14	101.43-138.80	---

Table 4 Physical Properties of Thermoplastics

POLYMER/COMPOSITE	OI AT 23°C
ACRYLONITRILE BUTADIENE STYRENE (ABS)	34
9,9 BIS (4-HYDROXYPHENOL) FLUORENE POLYCARBONATE-POLY (DIMETHYL SILOXANE) BLOCK POLYMER (BPFC-DMS)	47
POLYETHER SULFONE (PES)	40
POLYARYL SULFONE (PAS)	36
POLYPHENYLENE SULFIDE (PPS)	50
EPOXY	23
PHENOLIC	25
POLYAROMATIC MELAMINE	30
BISMALEIMIDE	35
40% EPOXY/181 GLASS CLOTH	27
40% PHENOLIC/181 GLASS CLOTH	57
40% POLYAROMATIC MELAMINE/181 GLASS CLOTH	42
40% BISMALEIMIDE/181 GLASS CLOTH	60

Table 5 Oxygen Index for Polymers and Composites

POLYMER	POLYMER STRUCTURE	T _g °C
EPOXY		335
PHENOLIC		525
BISMALEIMIDE		425
POLYAROMATIC MELAMINE		450

Table 6 Typical Chemical Structure of Thermoset Resins

THERMOPLASTICS	THERMOSETS	
ACRYLONITRILE BUTADIENE STYRENE	EPOXY	LOW
POLYARYL SULFONE	POLYAROMATIC MELAMINE	
POLYETHER SULFONE	PHENOLIC	
9,9 BIS (4-HYDROXYPHENOL) FLUORENE POLYCARBONATE-POLY (DIMETHYLSILOXANE)	BISMALEIMIDE	
POLYPHENYLENE SULFIDE		HIGH

Table 7 Relative Fire Resistance of Polymers