DESIGN, SYNTHESIS, AND DEVELOPMENT OF NEW TRANSPARENT

POLYMERS FOR MILITARY APPLICATIONS

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ABSTRACT

NASA's program on transparent polymers for military aircraft canopies has been expanded and now includes a wide range of performance properties not identified previously. The three major areas that are considered are: (1) fire resistance — particularly in relation to carrier deck fires; (2) utility barriers to high energy radiation; and (3) ballistic tolerance. In this paper attention is focused on the role of polycyclic aromatic transparent polymers, in solving these three classes of problems, and on the enunciation of the underlying thermochemical mechanisms we have found useful in the case of the epoxy derivatives. A new material based on a methylolated epoxy system is discussed. Relative to the third problem area, ballistic tolerance, it was noted that monolithic epoxy panels were quite brittle and laminate structures were considered. The results of ballistic tests on these laminates is discussed together with research into the molecular configuration of some polycarbonate structures.

INTRODUCTION

The NASA program on transparent polymers for military applications is an outgrowth of research into the development of fire-resistant materials that would provide increased protection of passengers involved in civilian aircraft fires. The work, jointly sponsored by NASA and the Joint Technical Coordinating Group for Aircraft Survivability (JTCG/AS), has been addressing aircraft cockpit protection against carrier deck fires and crash fires. This paper describes some of the current research and development work as well as the testing in order to permit comparison of the product of this research and development effort with state-of-the-art materials. It is shown that material developed for fire-resistance can, in general, be considered as hardened against other thermal threats as well.

Some related problems and early work were identified in a previous NASA paper presented at the 1973 Conference on Transparent Enclosures; some of the desired performance characteristics for advanced aircraft canopies, taken from reference 1, are shown in Fig. 1. The glass transition temperature (T_g) has been used as a guide in estimating the retention of mechanical strength at elevated temperatures. Polymers that have glass transition temperatures above 260° C have been considered suitable for use in the aerodynamic heating environment. In general, the higher the glass transition temperature the greater the tensile strength one can expect at the service temperatures. Because of its excellent impact resistance, polycarbonate was chosen as the starting point of an investigation into the feasibility of achieving improved thermal performance while retaining good mechanical properties (Ref. 1). None

of the state-of-the-art transparent polymers perform very well when exposed to fuel fire thermal environments.

Parker and Winkler (Ref. 2) have shown that the primary thermochemical anerobic char yield as measured from a thermogravimetric analysis (TGA) bears a direct relationship to the number of multiply bonded aromatic rings present in the initial polymer. Fish and Parker (Ref. 3) have shown that the resistance of a polymer to the intrusion of thermal radiation, as in the case of a JP-4 fuel fire, is critically controlled by aromatic nature of the initial polymer or its primary pyrolysis residue.

Figure 2 shows the effect of aromatic structure on the thermochemical char yield of transparent polymers as well as on some other aromatic polymers. Here, the char yield at 700° C versus the number of moles of multiply-bonded aromatic rings per gram of polymers is plotted. Two polymers reported previously (Ref. 1), epoxyboroxine and phenolphthalein polycarbonate, are plotted with bisphenol-A polycarbonate for reference. This relationship has provided a convenient means of characterizing polymers for selection as transparent composite components.

BACKGROUND

Material Development

In the 1973 conference on transparent aircraft enclosures, we reported (Ref. 1) on the development of a transparency consisting of a laminate of an aromatic polymer made from a cured epoxy material and a polycarbonate. An epoxy resin was chosen as one of the components because its aromatic and cross-linked structure would contribute to the desired thermal stability. The particular epoxy resin system with which most of the work has been done is a purified version of the common diglycidyl ether of bisphenol A (DGEBA) using 5 pph of trimethoxyboroxine (TMB) as a curing agent. This material is known as EX-112. The cure cycle was a three step process: 3 hr at 80° C; 3 hr at 135° C; and finally 2.5-3 hr at 180° C. The cast plates of the partially cured resin could be made to the proper curvature and conformation by carrying out the final curing step on the properly shaped form. Many castings of the resin system have been made with little or no modification and laminated to commercially available bisphenol A polycarbonate plates to form windows for evaluation in aircraft (Ref. 4). The structure of EX-112 is shown at the top of Fig. 3.

It has been possible to cast the epoxy system directly onto the surface of existing polymethyl methacrylate (PMMA) canopies. In this case a lower final cure temperature was necessary because of the low melting-temperature substrate PMMA, but a longer cure time at the lower temperature apparently permitted an adequate cure since little loss in the final thermal or physical properties was observed.

An analytical study of the epoxy-TMB system used has shown that about 2.3% of low molecular weight extractable oligomers (monomer, dimer and trimer), containing unreacted epoxide groups, remain in the cured resin (Ref. 5). These unreacted epoxide groups contributed 17.3% of the exothermic heat evolved at 430° C.

In 1973 (Ref. 1) we reported on the use of diglycidyl ether of bisphenol A as the only epoxy monomer used in the making of the polyether casting. This may indeed remain the preferred material because of its availability, color, cost, ease of use, and adequacy of performance. However, we have since investigated several other epoxide materials that we believed would have even better thermal properties because of their molecular structure. One of these was the "Apogen" (trademark) resins produced by the Metal and Thermit Company, Rahway, New Jersey. These are mono- and di-methylolated derivatives of DGEBA and were disclosed in a Belgian patent issued in 1965 (Ref. 6). The additional reactive methylol groups have the effect of easily forming crosslinkages between the resin molecules leading to a more dense network structure (Ref. 1). These resins were also cured with TMB, but are more difficult to process than the DGEBA/TMB system. These resins are more reactive and viscous. If one warms the resin to reduce the viscosity the increased reactivity due to the higher temperature, together with the additional reactivity due to the substituent, makes the casting process for thick sections quite difficult. However, there are ways of overcoming the difficulties: (1) by the use of reduced concentrations of TMB, (2) by the use of the less reactive tri-isopropoxyboroxine in mixture with the trimethoxyboroxine, or (3) by the use of mixtures of the Apogen resins with DGEBA. Not all of these combinations have been thoroughly investigated — especially as they relate to the thermal properties of the casting.

Using Apogen 101, the predominately monomethylolated derivative of DGEBA, preheated to 60° C to insure complete mixing with 5 pph of TMB, and otherwise proceeding as per the process with DGEBA, produced a clear transparent casting; this epoxide system is known as 4F9. In a thermogravimetric analyzer (TGA), this material exhibited an anaerobic char yield of 45% at 700° C compared to 34% obtained with the EX-112. In the T-3 thermal test the Apogen derived polymer (4F9) also showed improved thermal resistance — even longer time to burn-through and the black porous char that formed was stronger and had a finer, denser structure than that of the EX-112. The structure of 4F9 is shown as Epoxy No. 2 in Fig. 3.

The polyether resins made from the epoxy material are in general too brittle to be considered for use monolithicly in places where the impact of birds and other objects would be a problem. For that reason these castings were laminated to the high impact strength polycarbonate polymers as discussed in the 1973 paper (Ref. 1). The poly-bisphenol A carbonate material has been used in most of the prototype windows. It may be that this material will be the ultimate choice because of its availability, cost, color, and adequacy of performance, even though it lacks the required thermal properties (too low a melting point, glass transition temperature, and char yield).

Previously we chose as a promising candidate for our purposes the polycarbonate made from phenolphthalein, the structure of which is shown at the bottom of Fig. 3. In addition to the added aromatic ring, there is also a lactone ring capable of additional reactivity and crosslinking. The polycarbonate from phenolphthalein had good thermal and physical properties. There were problems, however, in the processing of this polymer. To make plates thick enough to use as windows required extraordinary procedures: molding of solvent plasticized polymer powder and the subsequent removal of the solvent

were extremely tedious tasks that required weeks of heat and vacuum treatment and often resulted in warped plates. High pressure and high temperature "forging" of the dry polymer powder required temperatures too near the decomposition temperature with the result that decomposition and premature cross-linking occurred. The lactone ring present in this material made the polymer somewhat too reactive for processing into window plates.

In a recent study (Ref. 7), other aromatic polycarbonates were made having as bisphenol monomers the structures shown in Fig. 4. All of these monomers have the pivotal carbon atom incorporated within a cyclic aromatic structure. The first compound, the 1,1-bis(hydroxyphenyl)-indan, was made but could only be obtained in a poor yield — 10 to 15%. The one below it in Fig. 4 — the 9,9-bis(4-hydroxyphenyl)-fluorene — was obtained in a considerably better yield of about 60%. There were problems in the synthesis of the other monomer materials and it was not feasible to spend more time in their study. The properties of these homopolymers are shown in Fig. 5.

Sufficient quantity of the indanyl bisphenol was obtained to make several batches of the polycarbonate; as shown in Fig. 6, it had very good physical and thermal properties. The polycarbonate made from the more accessible fluorenyl bisphenol was also made, but there were problems with its solubility in the polymerization mixture. This resulted in inherent viscosities of 0.18 to 0.26 (0.5% in tetrachloroethane) and films made from the material were cloudy and brittle. The addition of catalytic amounts of pyridine increased the inherent viscosity to 0.9; films made of this material were tougher but still somewhat cloudy.

Extensive work with this fluorenyl-bisphenol polycarbonate elsewhere has shown similar problems (Ref. 8). Gel permeation chromatograms were bimodal showing considerable low molecular weight oligomers to be present. Some of these can be removed by extraction with acetone. The thermogram (TGA) of the poly fluorenyl-bisphenol carbonate shows a T_d of 400° C and an anerobic char yield at 800° C of 61%. In air the onset of oxidative weight loss is at 300° C with the T_d again about 400° C and a surprizing char yield in air of 31%. The resulting charred foam had good integrity.

The excellent thermal properties of this fluorenone-bisphenol based polycarbonate, together with the ease of obtaining the monomer makes this an attractive candidate for further development. Because of difficulties in the polymerization of this material alone, however, attempts were made to make copolycarbonates of fluorenyl-bisphenol with both bisphenol A and with phenolphthalein. It was found that these co-polymerizations proceeded more readily.

Figure 7 shows the results of some of this copolymer work. The decomposition temperature and anerobic char yield are given for four copolymers. The anerobic char yields of these copolymers were quite satisfactory and the films of them were clear and tough. A quantity of the monomers has been obtained in order to make the larger amounts of the several copolymers that are necessary to permit further evaluation of the processability, and thermal and mechanical properties of these materials.

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LABORATORY TESTS

The tests performed on the materials followed standard ASTM, Mil Spec, or Federal test procedures wherever possible to enable comparison with handbook values of materials not included in the program; however, some tests are not standard types. These include the Ames T-3 test for "crash fire endurance," the high energy laser penetration tests, and ballistic tolerance tests.

The Ames T-3 thermal test facility is a JP-4 fuel-fired unit where the heat flux and thermal-chemical environment of a crash fire can be duplicated in a controlled manner. The results reported can either be burn-through or temperature-time history of the unexposed surface of materials undergoing test.

Using a high energy CO_2 laser the resistance of the materials was determined by exposing them to known flux rates for precise periods of time.

The ballistic impact tolerance of the materials was determined by observing their behavior in response to the impact of ball and armor piercing ammunition, and to the impact of fragments simulating nearby bursts of high explosive (H.E.) shells.

In the tables of properties the following material abbreviations are used:

1. acrylate, PMA

2. stretched acrylic, SPMA

3. bisphenol A polycarbonate, BPAPC

4. epoxy EX-112, 112

5. epoxy EX-4F9, 4F9

6. phenolphthalein polycarbonate, PHPC

7. polyarylsulfone type A, PASA

Physical Properties

The physical properties measured were tensile strength and modulus, flexural strength and modulus of elasticity, Izod impact strength and hardness. Tests of tensile properties were performed in accordance with ASTM D-638. The samples had a gage length of 2.54 cm and a cross section of 0.6 cm by specimen sheet thickness of approximately 0.6 cm. The tensile strength is the ultimate strength and tensile modulus was determined at 1.5% offset from the straight-line portion of the tensile stress-strain plot. The flexural tests were performed in accordance with ASTM D-790, method I, where the sample rests on two supports with a span of 10 cm, and is centrally loaded at a uniform rate. The modulus of elasticity is determined from the straight-line portion of the stress-strain curve during the maximum loading rate on the outer surface of the specimen.

The Izod impact strength tests, as prescribed by ASTM D-256, were performed on both virgin materials and on weathered materials — the weathered samples having been exposed to 500 hr (250 cycles) of continuous xenon light, with water spray for 18 min of each 2-hr cycle. The 6000-W xenon light with borosilicate filters has a spectrum similar to direct noon summer sunlight at Chicago, Illinois. In the impact test, notched samples were impacted by a weighted pendulum and the bending moment per width of specimen (width of notch) recorded. The hardness tests were performed on a Rockwell hardness tester and are shown both in "M" and "P" scales due to the wide range of hardnesses of the materials tested (ASTM D-1706). (The "M" scale uses a 6.35-mm steel ball penetrator and a major load of 100 kg; the "P" scale uses a major load of 150 kg.) Another indication of the relative hardness of the material is shown by abrading the surface in a cyclic fashion with a standard abrasion material. For these tests, a Taber abrader was equipped with Calibrase CS10F abrasion wheels that were loaded with 1000-gm weights. The milligram weight loss per 1000 Hz is reported along with other physical properties in Table 1. Electrical properties of the dielectric constant and the dissipation factor are also noted in Table 1. These properties were determined by capacitive measurements at audio frequencies.

Optical Properties

Since we are considering transparent materials, their optical properties are of extreme importance. The color ranges from clear, water-white, through pale blue to amber. A more definitive color response may be seen by measuring the transmittance T, reflectance R, and thereby the absorptance A, with a scanning spectrophotometer. This was done for the virgin polymers, for polymers that had been abraded with CS10F wheels and 500-gm weights for 50 Hz, and for polymers that had been weathered for 500 hr of continuous xenon light and periodic water spray. Haze, the ratio of diffused transmittance to total transmittance times 100, is also reported. The index of refraction (n) was determined by ASTM D-542 using the microscope method. Here the apparent thickness is measured optically and the true thickness, mechanically. Therefore, $n = d_{true}/d_{apparent}$, the index of refraction.

These optical properties are shown in Table 2. The T and R values listed are the integrated areas under the spectrophotometric traces from approximately 380 to 1670 nm. Also listed are single-wavelength transmittance values T for approximately 450 nm (blue), 550 nm (yellow), and 650 nm (red). These wavelengths would generally bracket the peak visual efficiency range of the human eye.

Thermochemical Properties

Thermogravimetric analysis (TGA) was used to determine weight loss in terms of temperature when the materials were subjected to a uniform rate of heat rise. Although the materials may be heated in a variety of atmospheric gases, the most useful is dry nitrogen for determining the true thermodynamic char yield (ash residue) of a polymer. The results closely parallel the reaction of a polymer to large pool-fire heating situations. The TGAs of the material systems being reported on are reproduced on a common weight loss vs temperature plot (Fig. 8). From the TGA, the percent char yield at any

temperature, the decomposition temperature, and the rates of thermochemical reactions that occur can be determined. The heat distortion temperature is a measure of the point where a weighted penetrator enters or causes a deflection of the heated specimen.

Two other thermal properties were measured: the coefficient of linear expansion α, determined by the dilatometer method, and the thermal conductivity coefficient k, determined by guarded hot plate method. These and other thermochemical properties are listed in Table 3.

Fire Properties

A number of related tests are used to determine the fire properties of materials. Those reported here are: (1) flammability, in accordance with ASTM 635, in which the polymer is placed in a horizontal position, ignited by a bunsen burner, and note made of the rate of burning and occurrence of any self-extinguishment (SE); (2) limiting oxygen index (LOI) which is determined by placing the material vertically in a preset mixture of nitrogen and oxygen and measuring the concentration of oxygen required to burn the sample once it is ignited; and (3) fire barrier test, T-3, used to determine how long it takes for either burn-through to occur or for the backface temperature to reach a predetermined temperature when the sample is exposed to a fuel fire of 115 kW/m^2 heat flux. These values are presented in Table 4.

The laser-resistant properties are being determined at this time, with some preliminary results presented previously in a paper by Parker et al., at the Las Vegas Symposium on Transparent Aircraft Enclosures (Ref. 1). The epoxy systems, EX-112 and 4F9, and the phenophthalein polycarbonate material all show extremely good resistance to high-energy radiation transmission.

Current laser penetration data are presented in a classified paper by S. R. Riccitiello et al. (Ref. 9). Although reference 9 is classified, certain design data of a nonclassified nature can be presented here. As mentioned previously, one very good tool for use in the development of fire resistant ablative materials is the primary thermochemical char yield. This has been shown in the past (Refs. 2 and 3) to be related to the aromatic equivalance of the polymer, or the basic polymer structure. Although epoxies cannot be related in this way to the basic structure, the thermochemical char yield can be measured; a plot of char yield versus polymer recession rate when the polymer is exposed to high energy CO_2 laser impact is shown in Fig. 9. As can be seen, the polymer recession rate decreases as the char yield of the polymer is increased. It is this behavior that enables one to screen many materials that may be candidate laser barriers. The other materials shown on the plot of Fig. 9 are various formulations that were tried but that are not reported here.

APPLICATION TO MILITARY AIRCRAFT

Depending on the aircraft use, the particular characteristics desired in a new transparent polymer will vary considerably: A prime consideration for carrier-based aircraft might be fire resistance to burn-through; for transport aircraft it might be for crash-fire resistance. Helicopter designers might

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want transparent materials that resist abrasion and have ballistic tolerance to shattering and spallation. In advanced fighters, resistance to bird impact, aerodynamic heating tolerance, and resistance to rain erosion might be of prime interest. Obviously, all of these properties may not be acquired in a single monolithic polymer. For instance, the epoxy systems show, due to their increased hardness and higher cross-linking, a definite improvement in fire resistance, abrasion resistance, laser resistance, etc., but these very properties make them more susceptible to impact shattering and ballistic intolerance.

One might trade on a synergistic combination of materials to gain the needed improvement. One combination already being considered is a composite of epoxy on bisphenol A polycarbonate. An outer surface of epoxy would yield fire, laser, and abrasion resistance and the polycarbonate backing, due to its extreme ductility, would provide ballistic and shock resistance with spall retention. In this combination, the interlaminar adhesive used could be significant. Specimens have been fabricated using ethylene terpolymer (ETP), silicones, and others; the ETP interlayer has a disadvantage in that it burns readily but this problem might be overcome by using a silicone interlayer.

Laboratory specimens have been prepared with the epoxy cast directly on acrylate as mentioned earlier. These show a substantial increase in fire resistance with increasing thickness of epoxy (Fig. 10). This might lend itself to a ready retrofit capability for increasing the fire and laser resistance of existing aircraft canopies. However, as the thickness of directly cast epoxy increases, the resistance to thermal shock decreases due to a slight difference in thermal expansion coefficients. Further, the epoxy thus applied cannot be fully cured, as can be the EX-112 system, because the acrylate base cannot withstand the temperatures required. In this regard, another alternative would be to use a laminate of epoxy on acrylic in conjunction with a suitable interlaminar adhesive.

A contractor was selected to construct panels of epoxy cast on acrylics, laminated epoxy 112 (EX-112) and polycarbonate, and a laminated epoxy on acrylate (Ref. 4).

An extension of this contract is providing prototype canopies for the A-4 aircraft of the same basic construction as the laminated panels for large scale fire tests at the Naval Weapons Center, China Lake, California. The A-4 and its canopy section are shown schematically in Fig. 11.

In addition to the sliding canopy section, sets of the forward side panels of the canopy will also be fabricated for use in the tests.

BALLISTIC TOLERANCE TESTS

The Naval Surface Weapons Center was given the task, by the JTCG/AS, to evaluate ballistically the laminated transparent materials developed by Ames Research Center. These tests were conducted primarily to determine the damaged area produced by impacts of caliber .30 APM2 projectiles, caliber .30 ball M2 projectiles, and caliber .22 fragment simulating projectiles (FSP). In addition, estimated protection ballistic limits (PBLs) were obtained for caliber .22 FSP.

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Five transparent materials were evaluated:

1. EX-112 cast on acrylic

2. EX-112/30 mil ETP/acrylic

3. EX-112/10 mil ETP/polycarbonate

4. polycarbonate

5. stretched acrylic

The polycarbonate and stretched acrylic served as baseline materials for damaged area comparisons. The caliber .30 AP and ball projectiles were fired at approximately 2350 ft/sec, a velocity that corresponds to a range of 200 yards. The caliber .22 FSP had a striking velocity of about 1100 ft/sec. When damaged areas are compared, the materials performed in the following order of preference (smallest damage with polycarbonate).

1. polycarbonate

2. epoxy/30 mil ETP/acrylic

3. epoxy/10 mil ETP/polycarbonate

4. stretched acrylic

5. epoxy cast on acrylic

There was very little difference between materials (2), (3), and (4). The estimated PBLs for the NASA-Ames materials were equivalent to those of polycarbonate within experimental error. Behind the armor, debris was very low for polycarbonate and epoxy/10 mil ETP/polycarbonate, somewhat higher for the stretched acrylic and epoxy/30 mil ETP/acrylic, and very high for the epoxy cast on acrylic. The epoxy cast on acrylic targets suffered severe delamination against all impacting projectiles.

CONCLUSIONS

It has been shown that aromatic polymers offer extremely good resistance to thermal threats. These aromatic transparent polymers, discussed in terms of their use as canopy materials, provide resistance to fuel fire burn-through and laser resistance, and protection against high-temperature operating environments. Two high-temperature-resistant epoxy systems, EX-112 and 4F9, with reasonable impact properties, and a phenolphthalein polycarbonate having a high glass-transition temperature and impact resistance were developed as a result of this investigation. The results of this study suggest that a good compromise of properties can be obtained from a laminate of the epoxy-boroxine polymer sheet with the bis-phenol A polycarbonate. Epoxy-boroxine laminated to acrylate substrates shows a surprizing resistance to thermal effects and acceptable ballistic tolerance. This may offer a good compromise for retrofit of existing aircraft. Additional efforts to increase the environmental tolerance of these NASA developed transparent materials is currently underway.

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FIGURE TITLES

Figure 1. Desired combination of properties for advanced transparent enclosures.

Figure 2. Effect of aromatic structure on thermochemical char yield of transparent polymers.

Figure 3. Transparent polymers of interest.

Figure 4. Some proposed aromatic bisphenols.

Figure 5. Summary of polycarbonate properties — homopolymers.

Figure 6. Poly-bis(hydroxyphenyl)indane carbonate.

Figure 7. Copolymers of bis(hydroxyphenyl)fluorene.

Figure 8. TGAs of transparent polymers.

Figure 9. Recession rate versus char yield.

Figure 10. Backface temperature of acrylate-Ex 112 laminates exposed to JP-4 fuel fire.

Figure 11. Development of improved transparencies for fabrication of aircraft canopies.

(a) RETENTION OF FUNCTIONAL PROPERTIES (MECHANICAL STRENGTH AND TRANSPARENCY) WITH INTERMITTENT SERVICE FROM ~-50°C TO +260°C

(b) CRASHWORTHY TO 308 METER PER SECOND* IMPACT

(c) RESISTANT TO THE HEAT PENETRATION OF BURNING JET FUEL

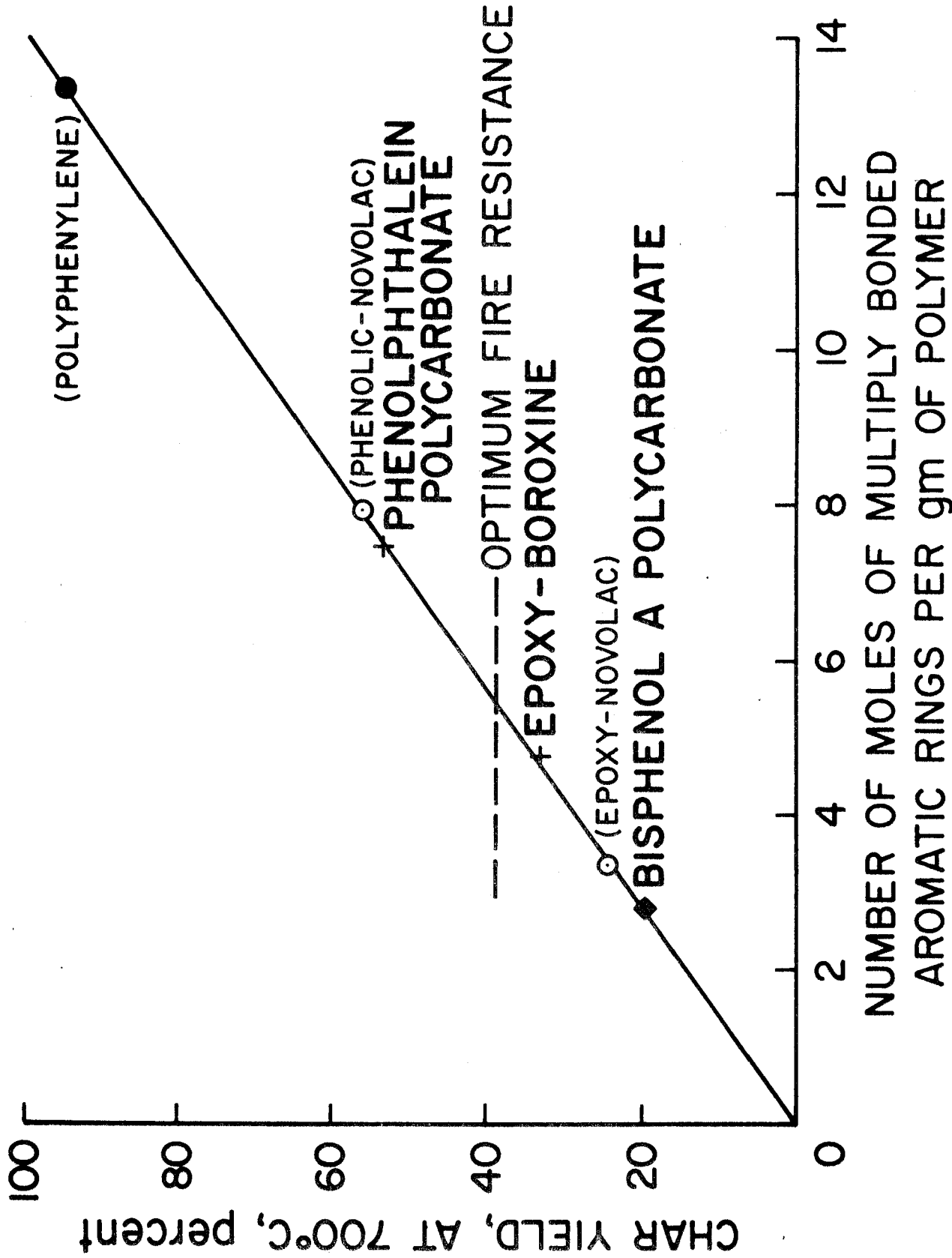(d) PROVIDES THERMAL PROTECTION FROM HIGH ENERGY COHERENT RADIATION

* 600 knots

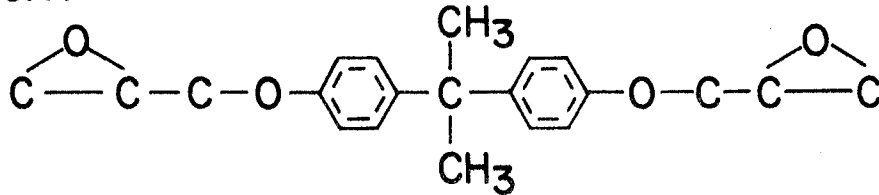Fig. 1

Fig. 2

CHAR YIELD, AT 700°C, percent

NUMBER OF MOLES OF MULTIPLY BONDED
AROMATIC RINGS PER gm OF POLYMER

(POLYPHENYLENE)

(PHENOLIC-NOVOLAC)
PHENOLPHTHALEIN
POLYCARBONATE

OPTIMUM FIRE RESISTANCE

EPOXY-BOROXINE

(EPOXY-NOVOLAC)
BISPHENOL A POLYCARBONATE

1. EPOXY

   a. CURED WITH TRIMETHOXYBROXINE-YIELD
      POLYETHER TYPE POLYMER

   b CURED WITH HEXAHYDROPHTHALIC ANHYDRIDE
      YIELD POLYESTER TYPE POLYMER

   c CURED WITH DIETHYLENE DIAMINE YIELDS
      POLYETHER TYPE POLYMER

2. EPOXY
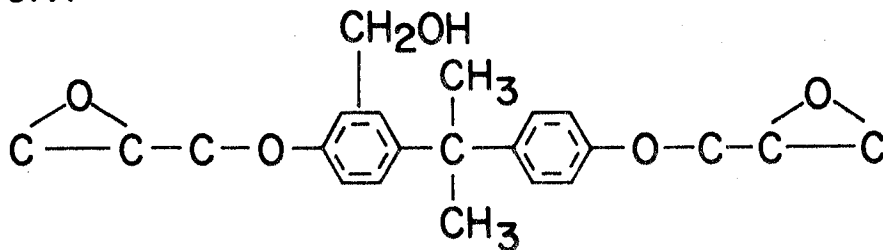
   80% MONO   20% di

   a. CURED WITH TRIMETHOXYBOROXINE-YIELD
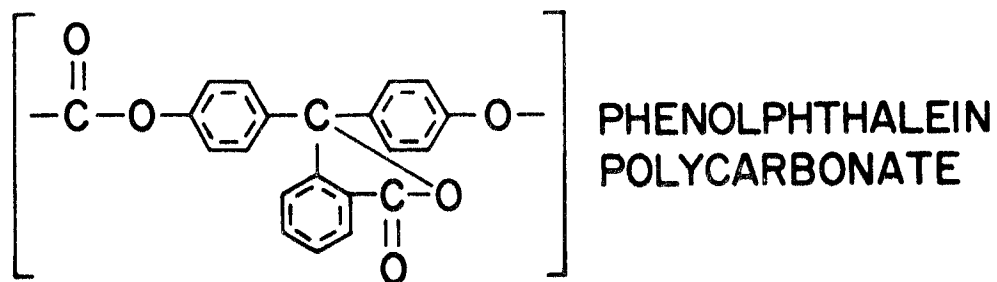   POLYETHER TYPE POLYMER

3. POLYCARBONATES

PHENOLPHTHALEIN
POLYCARBONATE

Fig. 3