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NASA Technical Memorandum 84494

SYNTHESIS AND CHARACTERIZATION OF A MELT PROCESSABLE POLYIMIDE

(NASA-TM-84494) SYNTHESIS AND
CHARACTERIZATION OF A MELT PROCESSABLE
POLYIMIDE (NASA) 32 F HC A03/MF A01

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INTRODUCTION

Linear aromatic polyimides are a class of polymers which are generally not processable via conventional thermoplastic or hot-melt techniques. This class of polymers is, however, exceptionally thermally stable and has high glass transition temperatures (1,2). They are also resistant to attack by common organic solvents (3).

Linear aromatic polyphenylene oxides and sulfides on the other hand are more easily processed than the polyimides (1), generally exhibit lower glass transition temperatures, and still have relatively good thermal stability although not equal to the polyimides (4). These systems also do not possess solvent resistance equal to the polyimides (5).

A novel linear aromatic polyphenylene ethersulfideimide has been synthesized which has some of the favorable characteristics of each parent system. The polymer has been molded, used as a resin, and cast into thin films. A limited characterization indicates this system can be processed via conventional thermoplastic techniques and may have a wide variety of applications.

EXPERIMENTAL

Preparation

Polymer - To form the polymer, 11.0000g of 4,4'-bis(3,4-dicarboxyphenoxy)diphenylsulfide dianhydride (BDSDA) and 6.2999g of 1,3-bis(amino-phenoxy)benzene (APB) were dissolved in 98.0g of bis(2-methoxyethyl)ether in a flask equipped with magnetic stirring. This solution was allowed to stir for one hour in order to build up molecular weight. The reaction is shown in Figure 1.

Molding Powder - The viscous polymer solution was poured into a mechanical blender containing distilled water. The contact with water caused the polyamide-acid to precipitate and the rotating blender blades chopped this material into a fluffy consistency. The solid polymer was isolated by suction filtration and allowed to air dry overnight.

The dried polymer was spread in a baking dish, placed in a forced-air oven and heated to 100°C. The polymer was held at this temperature for one hour to drive off residual water and solvent. The temperature was increased to 200°C to effect conversion of the amide-acid to the imide.

Unfilled Moldings - The polyimide powder was molded according to the following procedure. The imidized powder was placed in a matched-metal molding die which was preheated to 160°C. A pressure of 1.38-2.07 MPa (200-300 psi) was applied to effect consolidation. This temperature and pressure were held for one-half hour. The mold and molding were allowed to cool to approximately 100°C and the molding was removed. This molding was light brown and transparent when prepared in discs up to 0.635 cm (0.250 inches) in thickness.

Adhesive Bonds - Two sets of adhesive bonds were prepared at different heat-up rates. Duplicate 2.54 cm-wide strips of 0.127 cm (0.050 inch) thick titanium alloy were grit blasted with 120 mesh aluminum oxide, washed with methanol, and treated with Pasa Jell 107*. These strips were washed with water and dried in a forced-air oven at 100°C for 15 minutes. Each strip was coated with the polyamide-acid solution. A piece of woven glass cloth (0.01 cm

*Pasa Jell 107 is a commercial product of American Cyanamid Co. (Use of trade names or manufacturers does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.)

thickness) was laid into the wet polymer on one of the titanium panels. The coated panels were allowed to air dry for approximately one hour and then placed in a forced-air oven and heated to 160-275°C in order to drive off solvent and to convert the polyamide-acid to the polyimide. Imidization occurs at temperatures above 160°C and the degree of conversion is a function of time and temperature. An example of a cure is as follows:

1 hour at 100°C

1 hour at 200°C

1 hour at 275°C

This treatment was repeated several times on the panel with the glass cloth in order to build up sufficient adhesive thickness for bonding.

The panels were then overlapped according to ASTM D1002-72 and bonded in a hydraulic press under 2.07 MPa (300 psi) pressure. This sample was heated to 316°C at 5°C/min and held at temperature for fifteen minutes. The bonded specimen was allowed to cool to 100°C before removal from the press.

In order to achieve a higher heat-up rate another pair of adherends were placed into the press which had been previously heated to 343°C. The temperature rise on these panels was monitored, as in the previous case, using a thermocouple which was spot-welded at the edge of the bondline. This bonding procedure afforded an average 22°C/min heat rise to 316°C. A bonding pressure of 2.07 MPa was also used for this sample.

Film - A 15% solution of BOSDA/APB in diglyme was used to cast a 381µm thick wet film on plate glass using a doctor blade. The film was then cured in an air oven as follows:

1 hour at 100°C

1 hour at 200°C

1 hour at 300°C

The cured film, approximately 40 μ m thick, was removed from the glass for future testing.

Characterization Methodology

Flow Properties - Melt flow properties for BDSDA/APB in the range 250°C-350°C were determined using a capillary rheometer (Instron Model 3211) (6). The capillaries used had length-to-diameter ratios of 33 and 66; therefore no end corrections were required. Melt flow properties were not measured below 250°C because the pressure needed to force the polymer through the capillary was greater than the rheometer load cell was capable of measuring. Above 350°C, the flow measurements became very erratic.

Physical Properties - Water absorption at ambient temperature was determined using four 0.635 cm x 0.635 cm x 3.493 cm bar samples cut from molded stock. The samples were weighed and oven dried in air for 24 hours at 100°C. They were then cooled to ambient in a desiccator and reweighed. They were immersed in distilled water for 24 hours, removed and blotted dry and reweighed. Samples were reimmersed for an additional 48 hours, removed and blotted dry and again weighed.

Density determinations (gms/cm³) were made on two molded discs 6.350 cm diameter x 0.318 cm thick and the average value reported.

Inherent viscosity measurements were made using a Cannon-Ubbelohde viscometer in a 35°C water bath. A 10 ml solution of 0.5% solids in DMAc was made and filtered. The average of five runs of this solution was reported.

The number-average molecular weight (\bar{M}_n) and weight-average molecular weight (\bar{M}_w) were determined at room temperature for the polyamide-acid

dissolved in tetrahydrofuran.* A Knauer Membrane Osmometer was used for the \bar{M}_n measurements and a Brice-Phoenix Light Scattering Photometer for the \bar{M}_w measurements.

Mechanical Properties - All mechanical properties were determined at room temperature using an Instron Testing Machine Model TT-C.

The flexural strength and modulus of three samples 0.635 cm x 0.254 cm x 3.175 cm, cut from a molded disc, were run in three-point bending using a span of 2.54 cm and a crosshead speed of 0.127 cm/min. Due to a lack of material, these samples had a span-to-depth ratio of 10 and not 16 as specified by ASTM Standard D 790-71. The average of three samples was reported.

The compressive strength was determined for four samples nominally 0.645 cm x 0.709 cm x 1.224 cm. They were run at 0.127 cm/min crosshead speed and the average value reported. ASTM Standard D 695-69 was used as a guide, but due to lack of material, sample size was decreased from the recommended ASTM standard size (1.27 cm x 1.27 cm x 2.54 cm).

The fracture energy value (G_{IC}), the opening mode strain energy release rate, was determined from two compact tension samples. The samples were machined from discs 5.715 cm diameter and nominally 0.127 cm thick, precracked, and run at a crosshead speed of 0.127 cm/min according to ASTM Standard E 399-78A. The average of two samples was reported.

The lap shear tests were performed in accordance with ASTM Standard D 1002-72. Four samples bonded at a low heating rate (5°C/min) and four bonded at a high heating rate (22°C/min) were measured for lap shear strength and the average value reported.

*Molecular Weights Determined by ARRO Laboratory, Joliet, Illinois.

Thermal Properties - The coefficient of thermal expansion was determined for the range 30°C-125°C using a DuPont Model 941 Thermomechanical Analyzer (TMA) operating in static air at a programmed heating rate of 10°C/minute. The glass transition temperature (T_g) was determined calorimetrically with a DuPont Model 990 Thermal Analyzer/Differential Scanning Calorimeter in static air at a programmed heating rate of 20°C/minute. The apparent glass transition temperature was determined using a DuPont Model 943 Thermomechanical Analyzer in static air at 5°C/minute, and also on a DuPont Model 1090 Dynamic Mechanical Analyzer under the same run conditions. Thermooxidative stability (weight loss vs. temperature) was determined using a Perkin-Elmer Model TGS-2 thermogravimetric system using a heating rate of 2.5°C/minute and an air flow of 15 cc/minute.

A thermomechanical spectrum of the polymer was obtained by torsional braid analysis (TBA). A glass braid was coated with a 10% diglyme solution of the polyamide-acid and precured to 300°C in air. This spectrum was obtained at a heating rate of 3°C/min in a nitrogen atmosphere.

Chemical Resistance - Six film samples approximately 40 μ m thick were measured for T_g (apparent) using a DuPont Model 941 Thermomechanical Analyzer (7). Each of the six samples was immersed in one of six commonly used solvents at room temperature for a period of 72 hours. Their physical condition was noted and they were removed and blotted dry. When completely dry, they were again measured for T_g (apparent) and any change was noted.

RESULTS AND DISCUSSION

Synthesis

BDSDA/APB was synthesized according to the reaction scheme as shown in Figure 1. Reaction of the monomers in the ether solvent diglyme produced a highly viscous polyamide-acid with an inherent viscosity of 0.66 dl/g. The thermal imidization of the polyamide-acid was carried out on the powder and resulted in a linear high molecular weight polymer which could be processed as a hot-melt thermoplastic due to its novel molecular structure. This ease of processing may be due to the incorporation of oxygen and sulfur linkages in the polymer backbone as well as the meta orientation of some of these linkages.

The polymer, after thermal imidization at 200°C in air, exhibited a relatively low T_g of 161°C as shown by the TBA spectrum (Fig. 2). Further imidization was accomplished by curing at successively higher temperatures. This further imidization was indicated by the increase in melt flow viscosity with increased cure temperature (Fig. 3).

Characterization

Flow Properties - The melt flow properties of a polymer are important in determining how it should be processed. Compression molding, milling, calendaring, extrusion, and injection molding are some of the more commonly used processing methods (8). The melt flow properties of BDSDA/APB were determined for the shear strain rate region (10^{-1} - 10^3 sec $^{-1}$) that these processing methods cover.

The stress as a function of strain rate data (Fig. 4) obtained using the capillary rheometer indicates the polymer to be pseudoplastic, a non-Newtonian shear thinning flow property, typical of molten polymers. The pressures associated with commercial molding presses correlate, generally, with stresses in the 10^4 - 10^5 Pa range. Most of the measured stresses for this polymer were within or exceeded this range. Consequently, high temperatures and low strain rates would be required for molding of BDSDA/APB (i.e., long times).

The apparent viscosity as a function of strain rate data at various temperatures (Fig. 5) is shown for the strain rates encountered in different industrial processes. The apparent viscosity was calculated by dividing the flow stress by the strain rate. As the strain rate was calculated from the volumetric flow data and was not corrected to obtain the wall rate, the viscosity is an apparent rather than a true viscosity (9). The BDSDA/APB polymer should be processable via compression molding and calendaring techniques. However, no conclusions can be drawn concerning the extrudability of the polymer above a strain rate of 135 sec^{-1} due to the stress and strain rate limitations of the rheometer in its present configuration.

Figure 6 compares the change in apparent viscosity with strain rate at the midrange processing temperature for this polymer (BDSDA/APB), commercially available Torlon*, and a typical widely used ABS resin (10). This comparison is made because no data have been generated on a linear aromatic polyimide system prior to this BDSDA/APB study. At low strain rates the BDSDA/APB exhibits a considerably lower melt viscosity (i.e., lower processing pressure) than Torlon or ABS resin and maintains this relationship even at the higher strain rates. This would indicate it to be a somewhat more easily processable material.

*Torlon is the registered trademark of an Amoco poly(amide-imide).

Curves of apparent viscosity as a function of temperature for selected strain rates corresponding to specific processing methods (Fig. 7) were constructed by cross-plotting the apparent viscosity-strain rate data. The curves show fairly uniform flow behavior with no apparent processing "windows". However, they do indicate a processing advantage, a well defined decrease in viscosity with increased temperature for the different processing strain rates.

A sample of BDSDA/APB was cured to a temperature of 200°C in air and allowed to cool to ambient. This procedure was repeated for three additional samples at 227°C, 255°C and 325°C. Figure 3 shows the apparent viscosity for these four samples at 320°C as a function of elapsed time at that temperature. The viscosity increase with increasing cure temperature may be due to either a continuing buildup of molecular weight of the polymer in the melt form or crosslinking taking place. The viscosity at the three lower cure temperatures does not assume a constant value until the polymer has been at its run temperature of 320°C for 40-55 minutes. The viscosity of the sample cured at 325°C did not change with time. Samples were then cured at 227°C and 255°C in nitrogen and the viscosities compared with the air cured values (Fig. 8). The viscosity for the 227°C nitrogen cured sample was 1.50 MPa-sec, which is 1.31 MPa-sec higher than that for the air cured. The viscosity stabilized after approximately 30 minutes at the 320°C run temperature. The viscosity for the 255°C nitrogen cured sample was 2.40 MPa-sec, which is 1.6 MPa-sec higher than that for the air cured. The viscosity of that sample was constant from the beginning of the 320°C run. Further studies would be required to determine the specific mechanisms that cause this effect.

Rubbery type flow of molten polymers during extrusion, especially in the 2×10^5 Pascal shear stress region, can cause distortion or "melt fracture" in the extrudate (11,12). The BDSDA/APB polymer exhibited this characteristic during melt flow determinations. Three samples of the polymer extruded at 300°C and generated at increasingly higher stresses were compared (Fig. 9). Extrudate (A) was formed at the lowest stress (3.57×10^5 Pa) and exhibited a relatively smooth uniform surface, indicative of a low degree of melt fracture. Extrudates (B) and (C) were generated at successively higher rates of 10.71×10^5 Pa and 17.01×10^5 Pa, respectively, and the melt fracture characteristics, as expected, became more pronounced. Although higher extrusion temperatures did not materially alter the melt fracture, lower molecular weight polymer might exhibit less melt fracture (11).

Physical Properties - Some physical properties for BDSDA/APB are listed in Table 1. The ambient moisture content of 0.15% by weight was determined by oven drying the sample in air for 24 hours at 100°C . A 72 hour water soak restored the sample to its original weight. An interim check at 24 hours showed the sample had regained one-half of its ambient moisture content. These values are an average of four samples taken at room temperature. The density was determined to be 1.34 g/cm^3 , typical for an amorphous, linear aromatic polymer. The inherent viscosity of 0.66 dl/g for the polyamide-acid reflects the molecular weight of the polyamide-acid. Because the polymer melts during subsequent processing to the imide form, there is a possibility for either an increase or decrease in its molecular weight. The imide form of the polymer is

insoluble, thus precluding the determination of its inherent viscosity. The number-average molecular weight (\bar{M}_n) for the polyamide-acid was 13,900. This was the average of nine determinations (three measurements for each of three solution concentrations). The weight-average molecular weight (\bar{M}_w) was 27,500 (four measurements for each of two solution concentrations).

Mechanical Properties - The mechanical properties determined for BDSDA/APB at room temperature are listed in Table 2. The flexural strength and flexural modulus of 75.1 MPa and 3.48 GPa, respectively, represent the average of three samples. A compressive strength of 153 MPa was the average for four samples. All three values compare favorably with engineering thermoplastics.

The critical rate of release of strain energy (G_{IC}) for this polymer was determined to be 4,100 J/m², a very high value compared to addition-curing polyimides which have G_{IC} values of approximately 100 J/m² (13). Even an elastomer toughened addition polyimide has been reported to have a value of only 387 J/m² (14).

Adhesive bonds were fabricated at heating rates of 5°C/min and 22°C/min using 6Al-4V titanium adherends. In both cases the room temperature lap shear values of 40.3 MPa and 43.4 MPa, respectively, were quite high compared to conventional polyimide adhesives (15).

Thermal Properties - Some of the more common thermal properties were determined for BDSDA/APB and are listed in Table 3. The coefficient of thermal expansion, $5.14 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$, is typical for state-of-the-art polyimides. The glass transition temperature (T_g), measured calorimetrically, was determined to be 160°C. The T_g (apparent) values for a dry molded sample and for one water soaked for 72 hours at room temperature were measured thermomechanically. The T_g of the water soaked sample, run wet, was 145°C, 7°C below that for the dry sample (152°C).

BOSDA/APB film exhibits thermooxidative stability similar to Kapton[®] film as shown in the thermogram Figure 10. The dynamic TGA curves obtained at a heating rate of 2.5°C/min and an air flow rate of 15 cc/min indicate that both films undergo essentially no weight loss below 400°C. The BOSDA/APB exhibits a 50% weight loss temperature of 561°C, only 19°C below that for the Kapton, while both films undergo complete degradation at approximately 610°C.

Chemical Resistance - The chemical resistance of BOSDA/APB thin film (40µm thick) to six common solvents was determined and the results listed in Table 4. Methyleneethyl ketone, cyclohexanone, xylene, and tricresylphosphate had no visible effect on the film and there was no change in T_g (apparent). Methylene chloride and cresol caused severe swelling and T_g measurements were not possible, although the methylene-chloride-soaked film did maintain sufficient integrity as a film to allow mounting in the T_g fixture.

CONCLUSIONS

A novel linear aromatic polyimide that has characteristics of both linear aromatic polyphenylene oxides and sulfides was synthesized and characterized. Thermal imidization of the polyamide-acid at 200°C resulted in a linear, high molecular weight polymer with a T_g of 161°C that could be processed as a hot-melt thermoplastic.

The polymer exhibited a considerably lower melt viscosity at its midrange processing temperature (300°C) than did either commercially available Torlon or a widely used ABS resin, both measured at their midrange temperature, 340°C and 200°C respectively. It also exhibited a major well defined decrease in viscosity with increased temperature, a definite processing advantage.

The density of 1.34 g/cm^3 is typical for an amorphous, linear aromatic polymer. The inherent viscosity of 0.66 dl/g and molecular weights of $13,900$ (\bar{M}_n) and $27,500$ (\bar{M}_w) were determined for the polyamide-acid because the imide form of the polymer was insoluble.

The G_{IC} value of $4,100 \text{ J/m}^2$ was very high compared to addition-curing polyimides which have values of approximately 100 J/m^2 or an elastomer toughened addition polyimide with a reported value of 387 J/m^2 . Room temperature lap shear values of 40.3 MPa and 43.4 MPa were quite high compared to conventional polyimide adhesives.

The coefficient of thermal expansion value $5.14 \times 10^{-5} \text{ }^\circ\text{C}^{-1}$ is typical of state-of-the-art polyimides. The T_g (apparent), 152°C , decreased by only 7°C when the polymer was subjected to a 72 hour water soak and tested wet. It is a very thermooxidatively stable polymer showing essentially no weight loss below 400°C and 50% weight loss at 561°C . Complete degradation did not occur before 610°C .

This novel polymer because of its melt-flow properties, high G_{IC} , chemical and moisture resistance, and thermooxidative stability exhibits considerable attractiveness as an engineering thermoplastic. In particular, due to the degree of melt-flow, this polyethersulfideimide shows potential as a composite matrix resin and as a hot-melt adhesive.

ACKNOWLEDGEMENTS

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TABLE 1. PHYSICAL PROPERTIES OF BDSDA/APB

WATER ABSORPTION ^a	
24 hr SOAK	0.07%
72 hr SOAK	0.15%
DENSITY ^b	1.34 g/cm ³
INHERENT VISCOSITY ^c	0.66 dl/g
MOLECULAR WEIGHT ^d	
NUMBER AVERAGE (\bar{M}_n)	13 900
WEIGHT AVERAGE (\bar{M}_w)	27 500

(a) BAR SAMPLES NOMINALLY 0.635 cm × 0.635 cm × 3.493 cm
CUT FROM MOLDED STOCK

(b) MOLDED DISKS 6.350 cm DIAMETER × 0.318 cm THICK

(c) SOLUTION OF 0.50% SOLIDS IN DMAC AT 35°C

(d) VALUES DETERMINED ON THE AMIDE ACID BY ARRO LABS

TABLE 2. MECHANICAL PROPERTIES OF UNFILLED BDSDA/APB

FLEXURAL STRENGTH	75.1 MPa (10.9 ksi)
FLEXURAL MODULUS	3.48 GPa (505 ksi)
COMPRESSIVE STRENGTH	153 MPa (22.2 ksi)
CRITICAL RATE OF RELEASE OF STRAIN ENERGY, G_{1c}	4100 J/ m²
LAP SHEAR STRENGTH (π/π)*	
LOW HEATING RATE (5°C/min)	40.3 MPa (5.85 ksi)
HIGH HEATING RATE (22°C/min)	43.4 MPa (6.30 ksi)

*** BONDED AND TESTED ACCORDING TO ASTM STANDARD D 1002-72**

TABLE 3. THERMAL PROPERTIES OF UNFILLED BDSDA/APB

COEFFICIENT OF THERMAL EXPANSION (30°C - 125°C)	$5.14 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$
GLASS TRANSITION TEMP (T_g), CALORIMETRIC	160°C
THERMOMECHANICAL T_g (APPARENT)	
TMA - DRY	152°C
WET	145°C*
DMA	159°C
DECOMPOSITION TEMP	561°C**

* 72 hr WATER SOAK, TESTED WET

** 2.5°C/min HEATING RATE, 15 cc/min AIR FLOW, 50% WT LOSS

TABLE 4. CHEMICAL RESISTANCE OF BDSDA/APB FILM*

SOLVENT	EFFECT	CHANGE IN T _g , (APPARENT), °C**
METHYLETHYL KETONE	NONE	NONE
CYCLOHEXANONE	NONE	NONE
XYLENE	NONE	NONE
TRICRESYLPHOSPHATE	NONE	NONE
METHYLENE CHLORIDE	SWELLED	NOT DETERMINED
CRESOL	SWELLED	NOT DETERMINED

* 40 μ THICK FILM

** THERMOMECHANICAL ANALYSIS OF SOLVENT-LADEN FILMS
SOAKED 72 hrs AT ROOM TEMPERATURE AND BLOTTED DRY

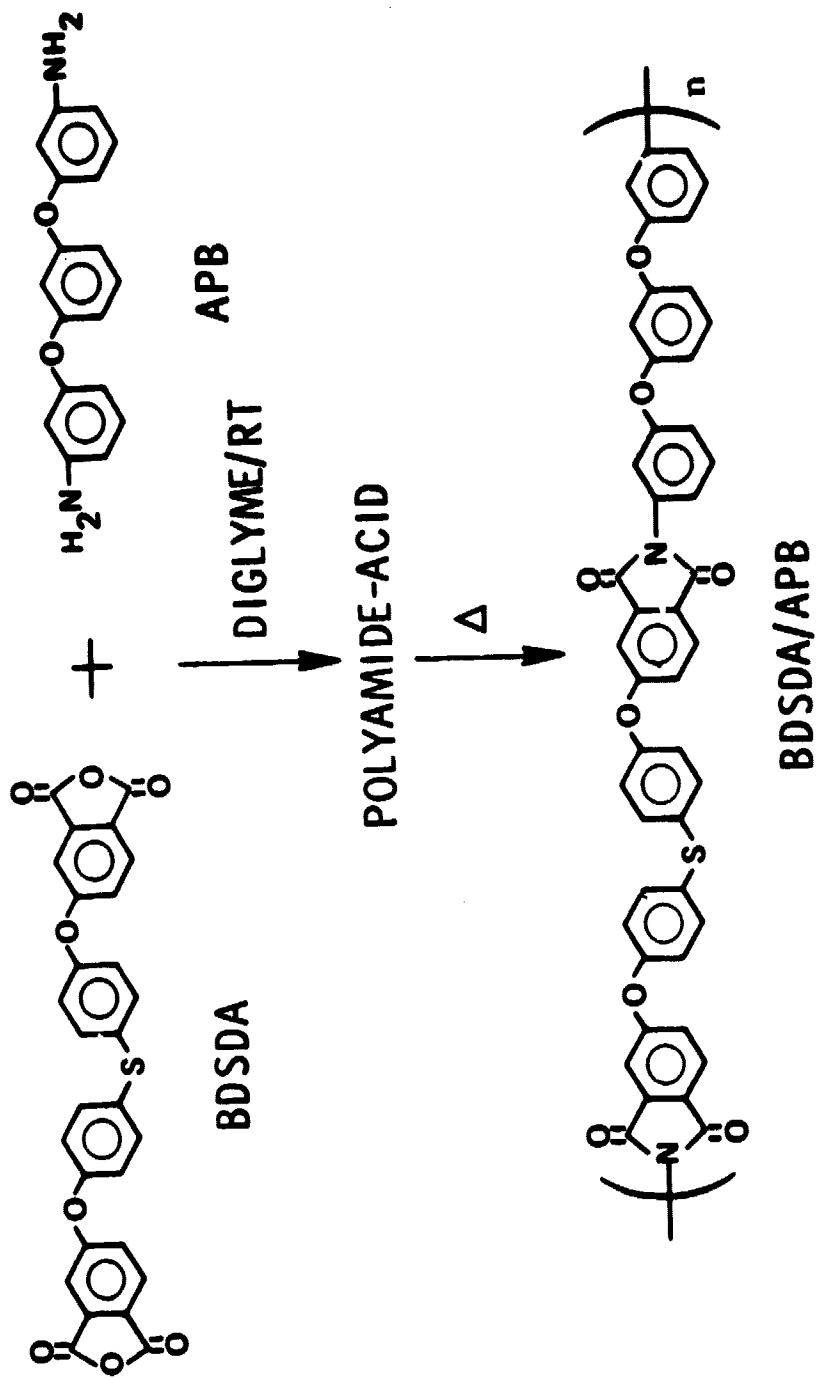


Fig. 1. Polymer Synthesis Scheme For BDSDA/APB.

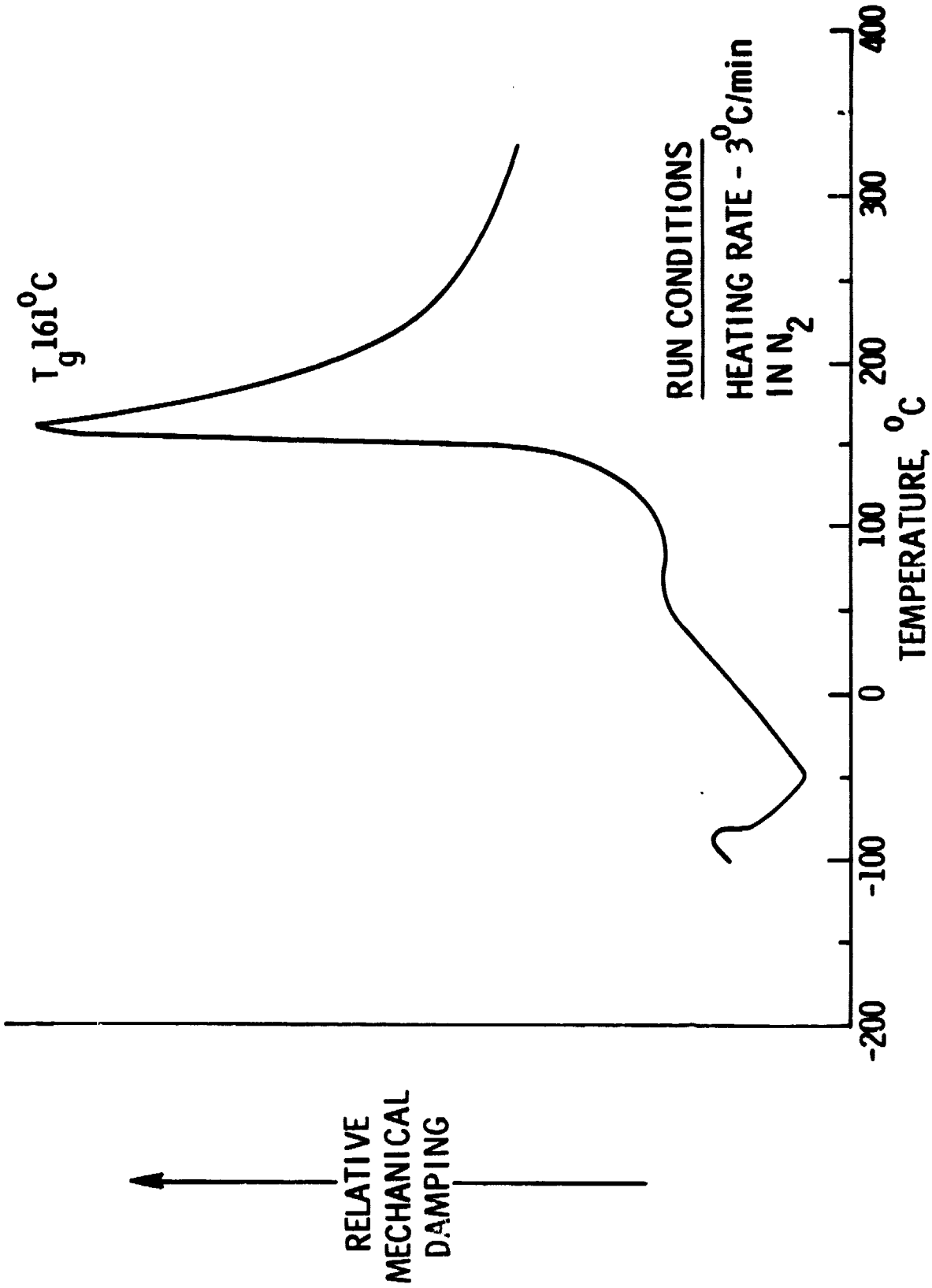


Fig.2. Torsional Braid Analyzer(TBA) Spectrum Of Imidized BDSDA/APB (Braid precured to 300°C in air).

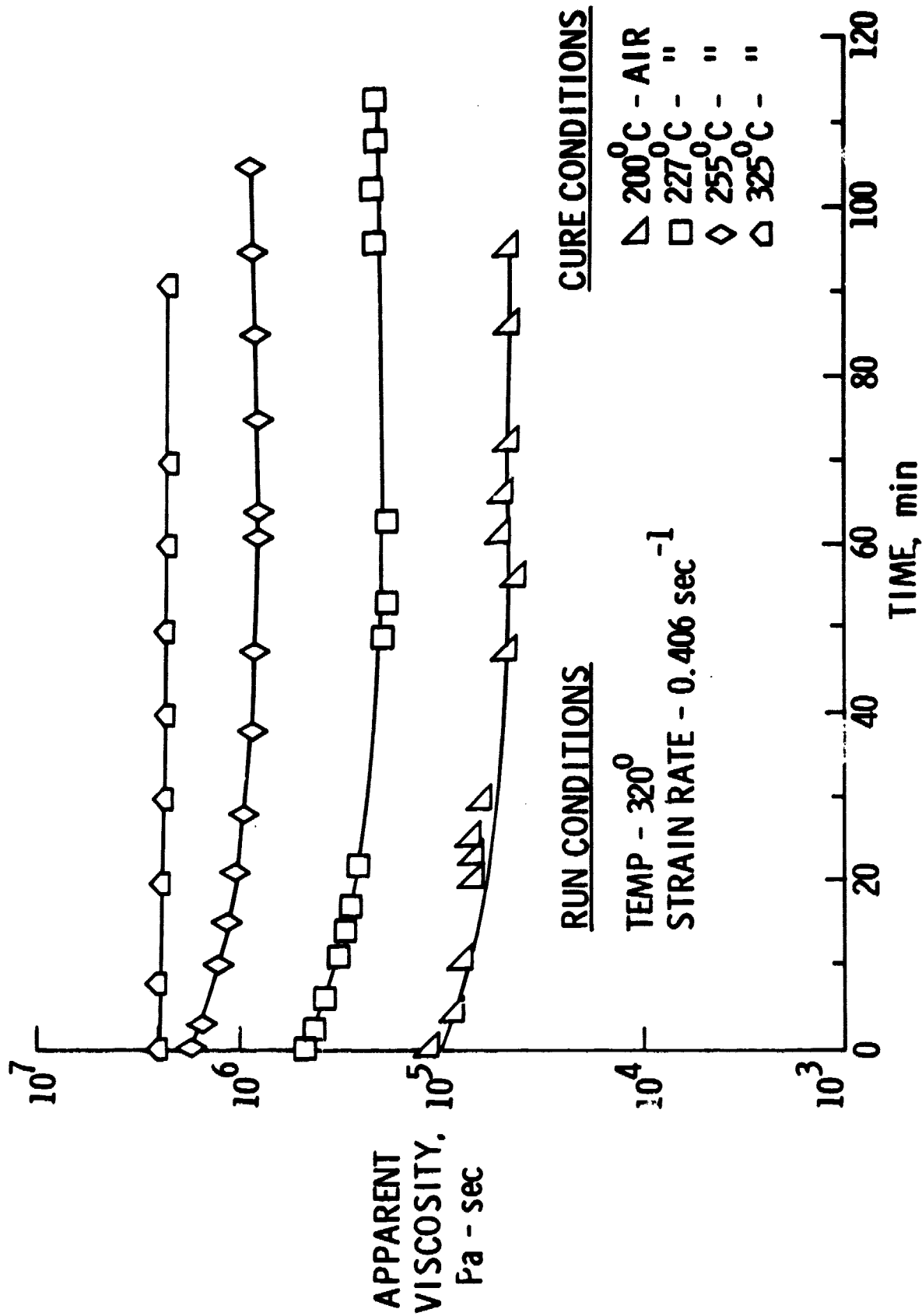


Fig. 3. Apparent Viscosity Of BDSA/APB As A Function Of Time And Cure Temperature.

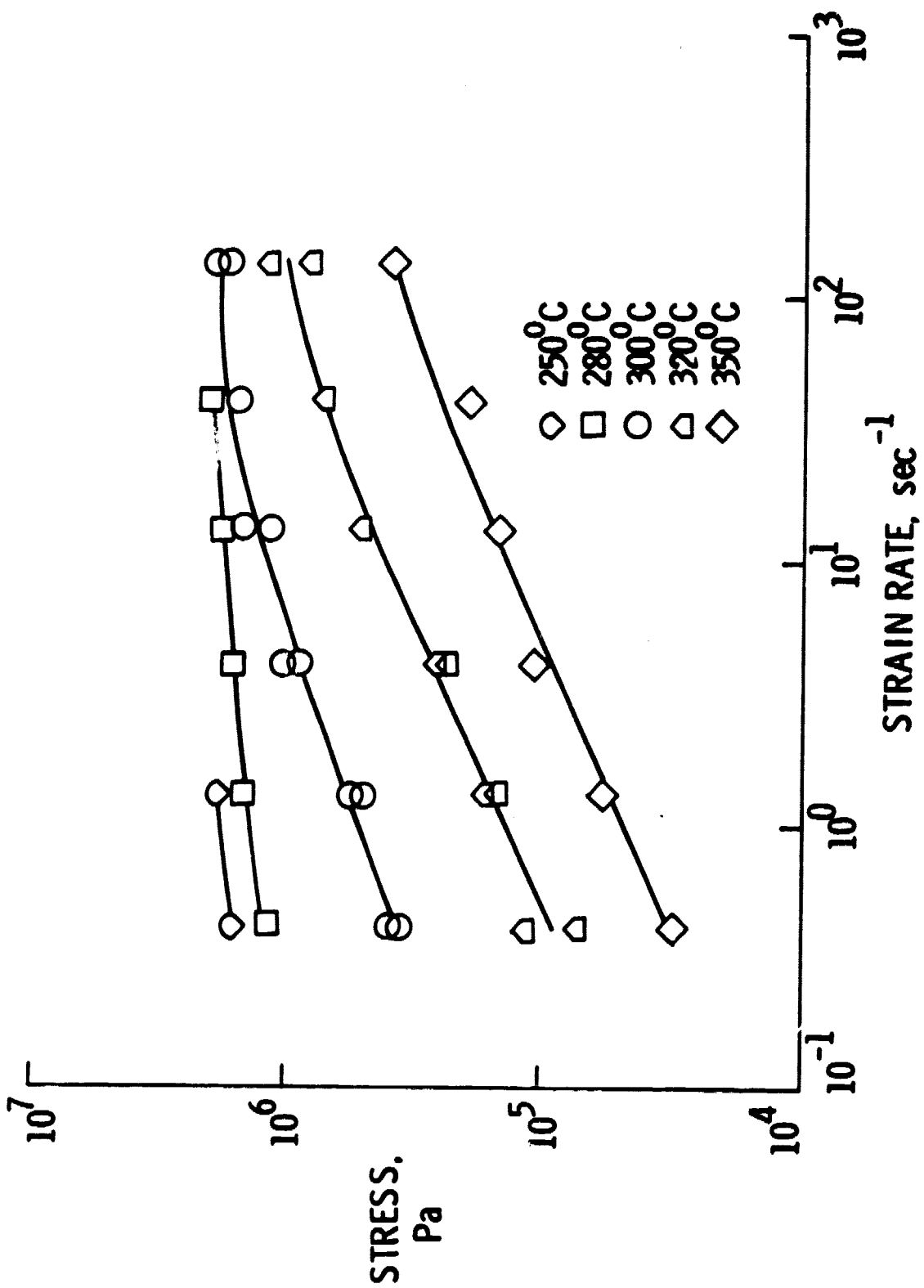


Fig. 4. Capillary Rheometer Stress Vs Strain Rate Data For BDSOA/APB In The 250°C-350°C Temperature Range.

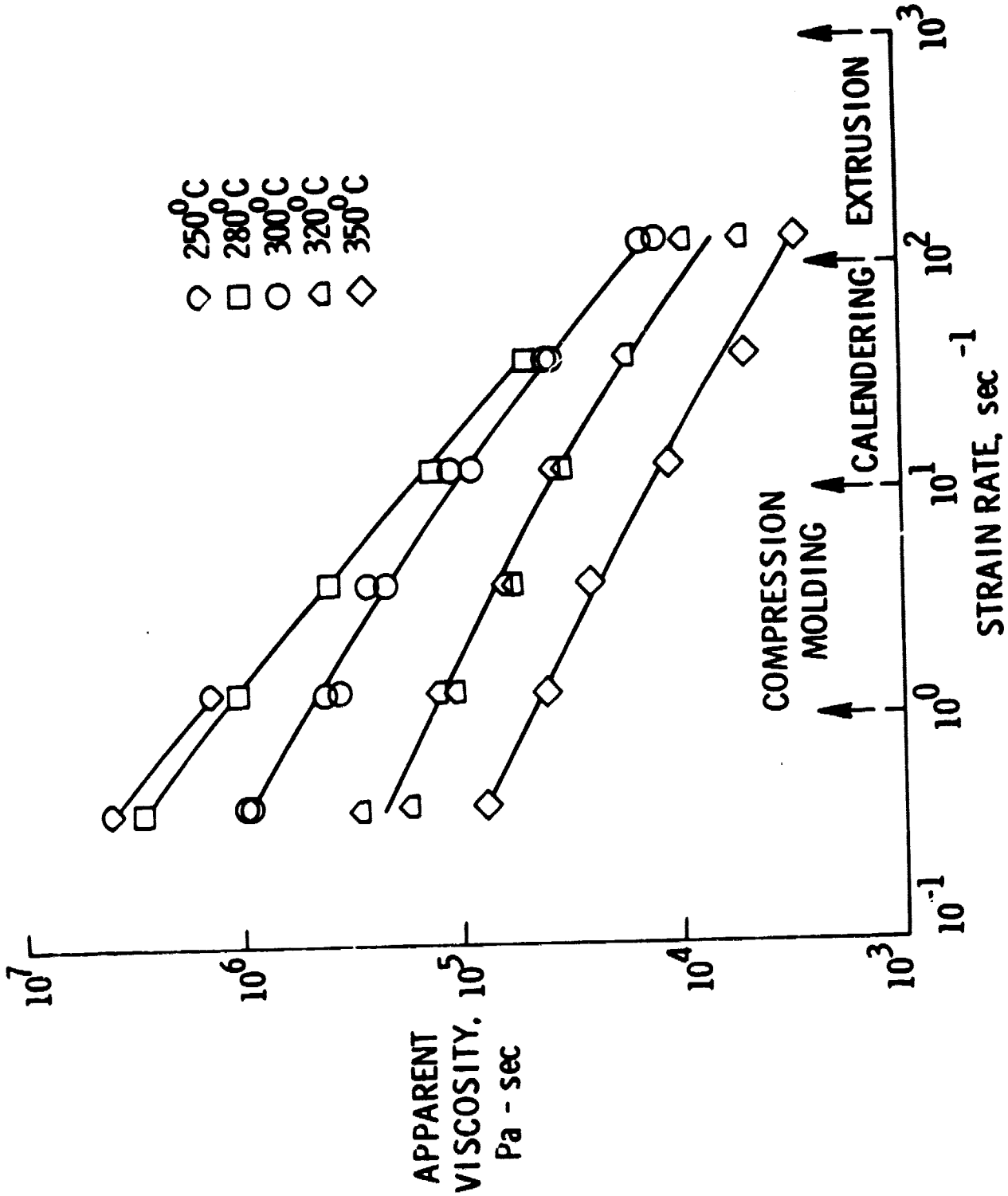


Fig. 5. Apparent Viscosity As A Function Of Strain Rate For BDSDA/APB In The 250°C-350°C Temperature Range.

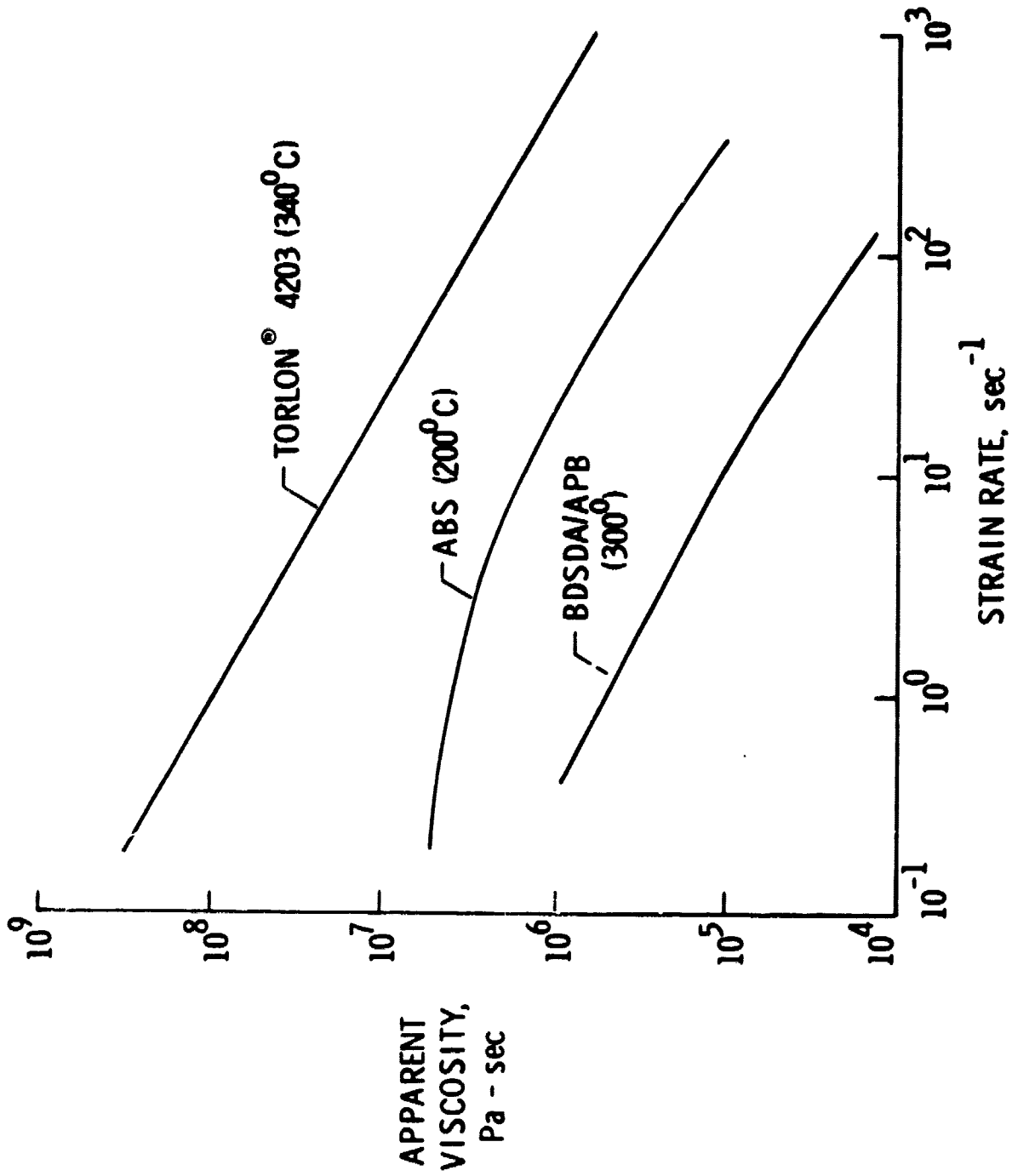


Fig. 6. Comparison Of Apparent Viscosity Vs Strain Rate Curves For BDSDA/PB, ABS And Torlon At Their Midrange Processing Temperatures.

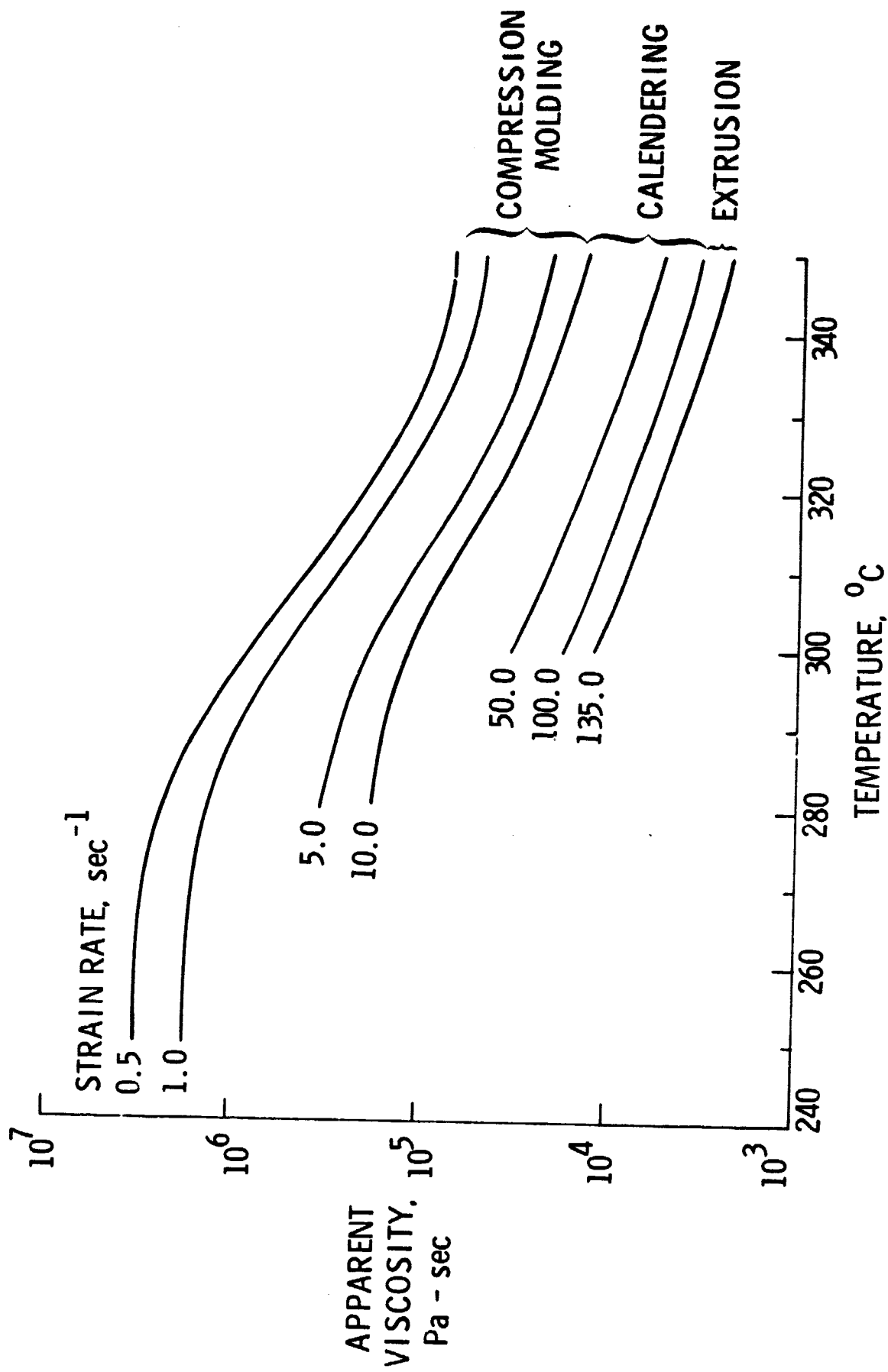


Fig. 7. Apparent Viscosity Vs Temperature At Various Strain Rates For BDSDA/APB For Specific Processing Methods.

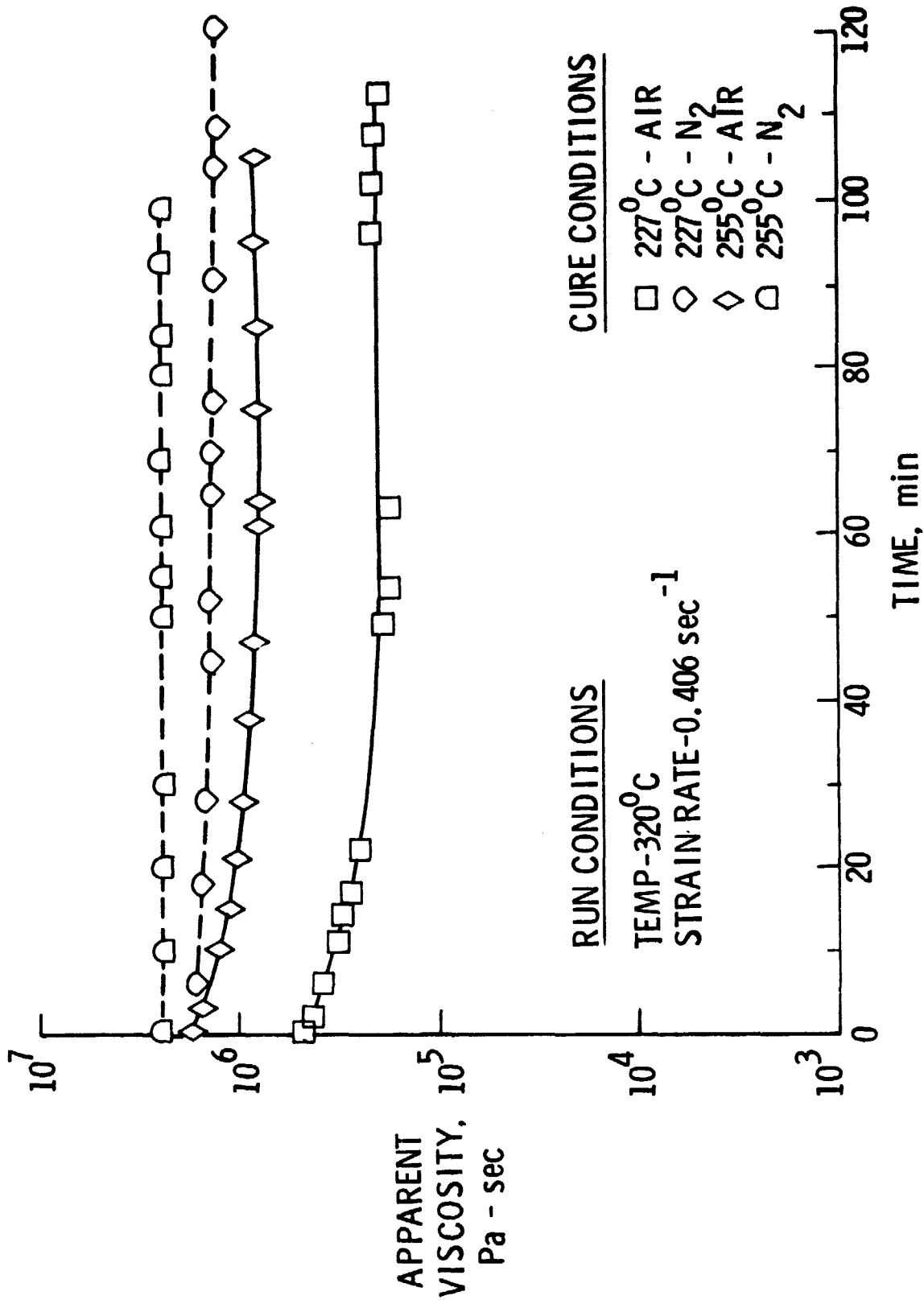


Fig.8. Comparison Of Apparent Viscosity Vs Time For Air And Nitrogen Cured BDSDA/APB.

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(a) $3.57 \times 10^5 \text{ Pa}$



(b) $10.71 \times 10^5 \text{ Pa}$



(c) $17.02 \times 10^5 \text{ Pa}$

Fig.9. Effect Of Increasing Stresses On The Melt Fracture Of BDSDA/APB Extrudate At 300°C.

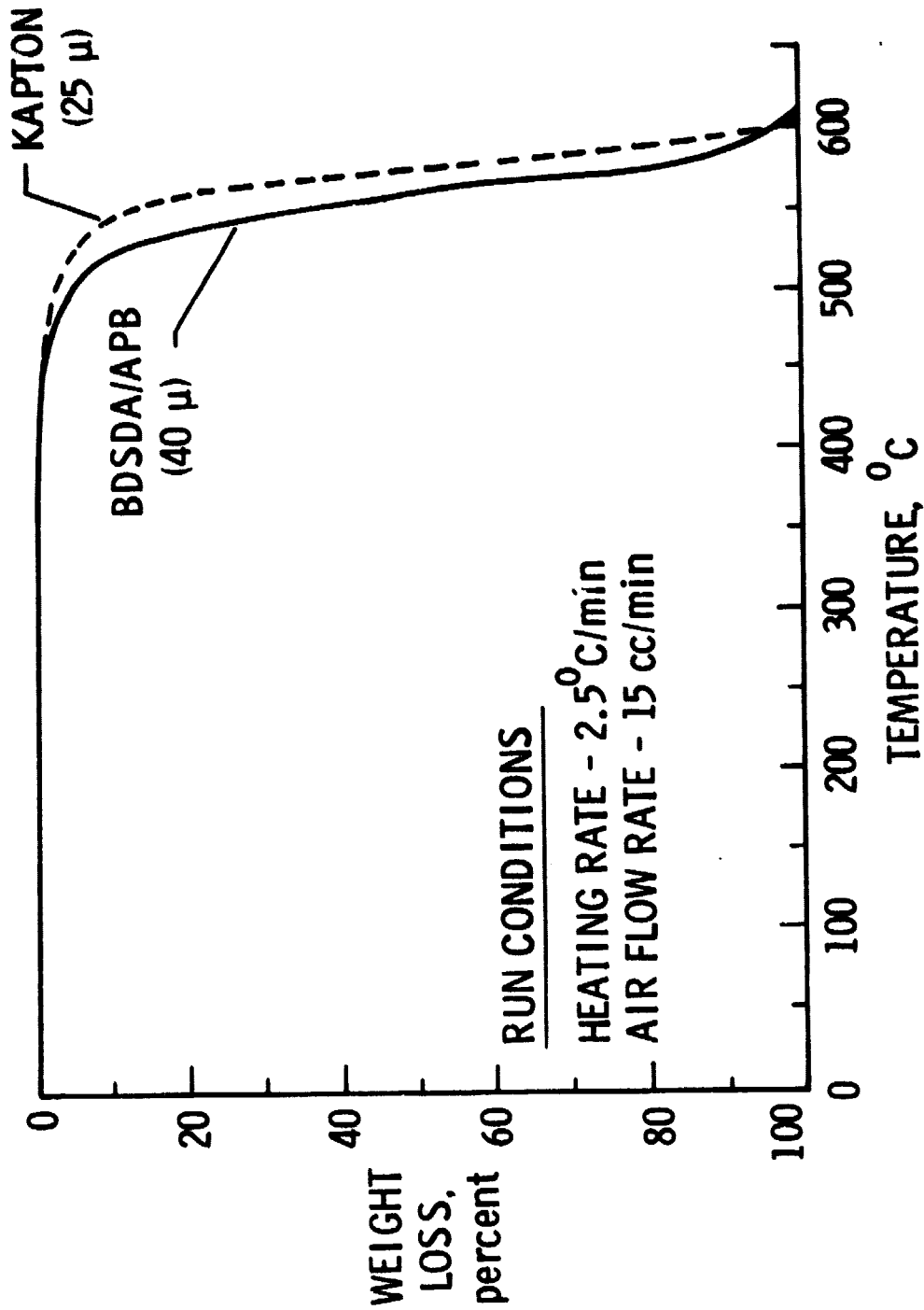


Fig. 10. Comparison Of Thermooxidative Stability Of BDSDA/APB And Kapton Polyimide Film.