NASA Technical Memorandum 84621

NASA-TM-84621 19830014153

SYNTHESIS AND CHARACTERIZATION OF A POLYETHERSULFONEIMIDE

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APRIL 1983

INTRODUCTION

Recently two linear aromatic polyimides have been shown to have consider-
able potential for processing via hot-melt techniques (1,2). The structures
for these systems are shown below.
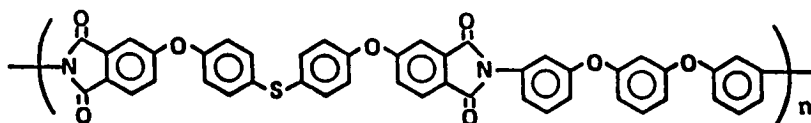

BDSDA/APB
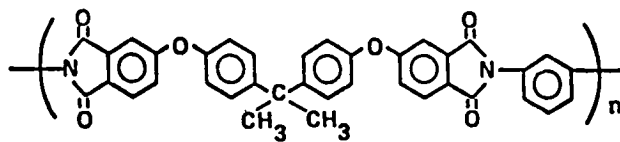
ULTEM*

There are two factors that appear to have produced this enhanced flow.
The first and perhaps most important factor is the separation of the imide
rings. In both structures the phthalimide moieties are separated by bisphenox-
ide units. This may have the effect of diluting the high associative forces
that have classically imparted the high glass transition temperatures to linear
aromatic polyimides (3). The second factor that is common to both systems is
the incorporation of diamines with meta-linkages. This technique has been
proven to lower glass transition temperatures in polyimides (4). The articu-
lation of both of these features gives the potential for some very processable
polyimides. The subject of investigation for this report are the two poly-
imides of the following structure.

*ULTEM is the registered trademark of a General Electric Polyetherimide.
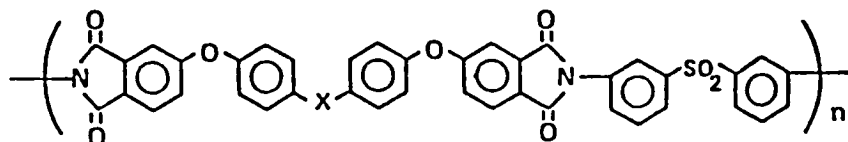
WHERE X IS —S— OR —SO$_2^-$

These two polymers were prepared and studied as part of a continuing program with the objective of developing hot-melt processable polyimides. The synthesis and characterization of these materials are discussed.

EXPERIMENTAL

Preparation

Polymer - To form the polymer, 0.1000 mole of 4,4'-bis(3,4-dicarboxyphenoxy)diphenylsulfide dianhydride (BDSDA) and 0.1000 mole of 3,3'-diaminodiphenylsulfone (DDSO$_2$) were dissolved in bis(2-methoxyethyl)ether at 15 percent solids 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.

The same procedure was used for the polymer made from 4,4'-bis(3,4-dicarboxyphenoxy)diphenylsulfone (BDSO$_2$DA) and DDSO$_2$. However, no appreciable buildup in molecular weight occurred during this step. A buildup was noted when the precipitated polymer was heated as outlined in the next subsection on molding powder.

Molding Powder - The viscous polymer solution was poured into a mechanical blender containing distilled water. The contact with water caused the

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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 and held for 1 hour to effect conversion of the amide-acid to the imide. Both systems fused into glassy solids at this stage. During this melt phase the $BDSO_2DA/DDSO_2$ system appeared to undergo further polymerization. This glassy polyimide was ground into powder form.

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 200°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. These moldings were light brown and transparent when prepared in discs up to 0.635 cm (0.250 inches) in thickness.

Film - A 15% solution of $BDSDA/DDSO_2$ in diglyme was used to cast a 381 μm 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

Since the $BDSO_2DA/DDSO_2$ did not build to high molecular weight during the solution polymerization, no films were prepared. The cured film of $BDSDA/DDSO_2$, approximately 40 μm thick, was removed from the glass for future testing.

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Characterization Methodology

Flow Properties - Melt flow properties for BDSDA/DDSO$_2$ and BDSO$_2$DA/ DDSO$_2$ at 375°C were determined using a capillary rheometer (Instron Model 3211) (5). The capillaries had length-to-diameter ratios of 33 and 66; therefore no end corrections were required.

Physical Properties - Water absorption at ambient temperature was determined using three bar samples of each material, each sample nominally 0.254 cm x 0.635 cm x 2.337 cm 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.

The density (gms/cm^3) was determined on a molded disc of BDSDA/DDSO$_2$ 5.715 cm diameter x 0.254 cm thick. Density determinations for the BDSO$_2$DA/ DDSO$_2$ were made on six bar samples nominally 2.663 cm x 0.639 cm x 2.739 cm cut from molded stock and the average value reported.

Inherent viscosity measurements were made on the polyamide-acid form of BDSDA/DDSO$_2$ 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 four runs of this solution was reported.

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

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of BDSDA/DDSO$_2$ dissolved in tetrahydrofuran.* A Knauer Membrane Osmometer was used for the $\overline{M}_n$ measurments and a Brice-Phoenix Light Scattering Photometer for the $\overline{M}_w$ 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 BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$, each sample nominally 0.635 cm x 0.254 cm x 3.175 cm and cut from a molded disc, were determined in three-point bending in accordance with ASTM Standard D 790-71. Due to a lack of material, these samples had span-to-depth ratios of 10-12 and not 16 as specified by the ASTM Standard. The average of four samples was reported.

The fracture energy value (G$_{Ic}$), the opening mode strain energy release rate, was determined for two compact tension samples of the BDSDA/DDSO$_2$ system. G$_{Ic}$ for the BDSO$_2$DA/DDSO$_2$ system was not determined due to a lack of material. 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.

Thermal Properties - The coefficient of thermal expansion was determined for the range 30°C-175°C using a DuPont Model 941 Thermomechanical Analyzer (TMA) operating in static air at a programed 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 (DSC) in static air at a programed heating rate of 20°C/minute. The apparent glass

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

transition temperature was determined using a DuPont Model 943 Thermomechanical Analyzer (TMA) in static air at 5°C/minute, and also on a DuPont Model 1090 Dynamic Mechanical Analyzer (DMA) under the same run conditions. The T_g (apparent) was also determined from a thermomechanical spectrum of BDSDA/ $DDSO_2$ obtained by torsional braid analysis (TBA). A glass braid was coated with a 10% diglyme solution of the polyamide-acid and precured to 250°C in N_2. This spectrum was obtained at a heating rate of 3°C/min in an air atmosphere. Thermooxidative stability (weight loss vs. temperature) was determined using a Perkin-Elmer Model TGS-2 thermogravimetric analysis system (TGA) using a heating rate of 2.5°C/minute and an air flow of 15 cc/minute.

Infrared Analysis - Infrared spectra of BDSDA/$DDSO_2$ and $BDSO_2DA$/ $DDSO_2$ in the 4000-400 cm^{-1} wavenumber region were obtained using a Nicolet Model MX-1 FTIR Spectrometer. Attenuated total reflectance technique (ATR) using a KRS-5 crystal was employed because the polymer samples were too thick for standard transmission measurements. Each spectrum is the average of between 100 and 500 individual scans.

Chemical Resistance - Six film samples of BDSDA/$DDSO_2$ approximately 40 μm thick were measured for T_g (apparent) using a DuPont Model 941 Thermomechanical Analyzer (6). 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) for any possible change.

RESULTS AND DISCUSSSION

Synthesis

BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ were synthesized according to the reaction scheme shown in Figure 1. Reaction of the BDSDA/DDSO$_2$ monomers in the ether solvent, diglyme, produced a viscous polyamide-acid with an inherent viscosity of 0.52 dl/g. As previously mentioned, the BDSO$_2$DA/DDSO$_2$ system did not build to a high molecular weight until the imidization step. The thermal imidization of both polyamide-acids were carried out on the powder and resulted in linear high molecular weight polymers which could be processed as hot-melt thermoplastics.

The polymers, after thermal imidization at 200°C in air, exhibited T$_g$'s of 209°C and 205°C, respectively, for the BDSDA and BDSO$_2$DA systems. Further molecular weight buildup was accomplished for the BDSO$_2$DA system by curing at 200°C as evidenced by an increase in the resistance of the polymer to being powdered by mechanical means.

Characterization

Flow Properties - The melt flow properties of a polymer are important in determining how it should be processed. Compression molding, milling, calendering, extrusion, and injection molding are some of the more commonly used processing methods (7). The melt flow properties of BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ were determined for the shear strain rate region (10^{-1}-10^3 sec^{-1}) that these processing methods cover.

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Capillary Rheometry was used to determine stress versus strain rate for BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ at 375°C (Fig. 2). These polymers were shown to be pseudoplastic, a non-Newtonian shear thinning 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 these polymers exceeded this range. They also exceeded the stresses required for BDSDA/APB as previously reported (1). Consequently, higher temperatures and low strain rates would be required for molding of BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ (i.e., longer times) as compared to BDSDA/APB.

The apparent viscosity as a function of strain rate at 375°C (Fig. 3) 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 (8). The BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ polymers should be processable via compression molding and calendering techniques. However, no conclusions can be drawn concerning the extrudability of the polymers above a strain rate of 40 sec^{-1} due to the stress and strain rate limitations of the rheometer in its present configuration.

Figure 3 also compares the change in apparent viscosity with strain rate of BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ at 375°C with BDSDA/APB at 350°C (1). At low strain rates BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ exhibit higher melt

viscosities (i.e., higher processing pressures) than BDSDA/APB and maintain this relationship even at the higher strain rates. This would indicate that they are somewhat less easily processable than BDSDA/APB.

Melt Fracture - As was the case with the previously reported BDSDA/APB (1), both BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ exhibited pronounced distortion when extruded at 375°C at shear stresses in the 2 X 10^5 Pascal region. This distortion, referred to as "melt fracture" (9,10), increased with increasing stress during the extrusion process. BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ at 375°C exhibited comparable melt fracture, and exceeded the melt fracture exhibited by BDSDA/APB at 350°C. Therefore, BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ might be more difficult to extrude.

Physical Properties - Physical properties for BDSDA/DDSO$_2$ and BDSO$_2$DA/ DDSO$_2$ are listed in Table 1 and show the polymers to be essentially identical relative to these properties. Ambient equilibrium moisture contents of 0.69% and 0.67%, respectively, by weight were determined by oven drying the samples in air for 24 hours at 100°C. A 72-hour water soak restored the samples to approximately two-thirds of their original weight. An interim check at 24 hours showed the samples had regained approximately one-third of their original ambient moisture content. These values are an average of three samples of each polymer taken at room temperature. The densities were determined to be 1.38 g/cm^3 and 1.37 g/cm^3, typical for amorphous, linear aromatic polymers. The inherent viscosity of 0.52 dl/g for the polyamide-acid form of BDSDA/DDSO$_2$ 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, as occurred in the $BDSO_2DA/DDSO_2$ system where an increase was evident. The number-average molecular weight ($\overline{M}_n$) for the polyamide-acid form of $BDSDA/DDSO_2$ was 8,500. This was the average of nine determinations (three measurements for each of three solution concentrations). The weight-average molecular weight ($\overline{M}_w$) was 12,100 (two measurements for each of four solution concentrations). Inherent viscosity and molecular weight were not determined for the $BDSO_2DA/DDSO_2$. In its polyamide-acid form it was soluble, but very low in molecular weight. In the imide form the molecular weight increased, but it was now insoluble thus precluding the determination of its inherent viscosity and molecular weight.

Mechanical Properties - The mechanical properties determined for $BDSDA/DDSO_2$ and $BDSO_2DA/DDSO_2$ at room temperature are listed in Table 2. The flexural strength and flexural modulus for the $BDSDA/DDSO_2$ of 27.1 MPa and 3.74 GPa, respectively, represent the average of three samples. The flexural strength and flexural modulus for the $BDSO_2DA/DDSO_2$ of 74.1 MPa and 3.36 GPa, respectively, also represent the average of three samples. The critical rate of release of strain energy (G_{Ic}) for $BDSDA/DDSO_2$ was determined to be 268 J/m^2. The low values for flexural strength and G_{Ic} for the $BDSDA/DDSO_2$ polymer are attributed to its low molecular weight (11).

Thermal Properties - Some of the more common thermal properties were de-termined for BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ and are listed in Table 3. The coefficients of thermal expansion 4.99 x 10^{-5}°C^{-1} and 4.96 x 10^{-5}°C^{-1}, respectively, are typical for state-of-the-art polyimides. The glass transition temperatures (T$_g$), measured calorimetrically, were deter-mined to be 209°C and 205°C respectively. The T$_g$ (apparent) values, deter-mined using TMA, were 211°C for both polymers. The DMA method established a T$_g$ (apparent) of 192°C for BDSDA/DDSO$_2$ and 200°C for BDSO$_2$DA/DDSO$_2$. BDSDA/DDSO$_2$ exhibited a T$_g$ (apparent) of 208°C when measured by TBA (Fig. 4).

The thermooxidative stability of BDSDA/DDSO$_2$ and BDSO$_2$DA/DDSO$_2$ film is shown in the thermogram in Figure 5. 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 the films undergo essentially no weight loss below 380°C. The BDSDA/DDSO$_2$ exhibits a 50% weight loss temperature of 588°C, 13°C above that for the BDSO$_2$DA/DDSO$_2$. The BDSDA/DDSO$_2$ film experienced complete degradation at approximately 650°C, 20°C above that for the BDSO$_2$DA/DDSO$_2$.

Infrared Analysis - The spectra between 4000 and 400 cm^{-1} were run on a BDSDA/DDSO$_2$ film cured in a nitrogen atmosphere, a molding of the air-cured BDSDA/DDSO$_2$, and a molding of the air-cured BDSO$_2$DA/DDSO$_2$. The spectra between 2000 and 400 cm^{-1} of the N$_2$-cured BDSDA/DDSO$_2$ film and the air-cured BDSO$_2$DA/DDSO$_2$ molding are shown in Figure 6. It is evident in this comparison that the two spectra are virtually identical. In Figure 7 the mold-ings from each system are compared. Again both systems appear identical. The logical explanation seems to be an oxidation of the sulfide group to the sulfone. The similarity of other physical properties which were

previously discussed might be attributed to this conversion. However, in another study of polyimides where a methylene bridge was converted to a carbonyl during cure, a crosslinking phenomena occurred which resulted in a higher T_g than was found for the corresponding carbonyl-containing polymer that had been unequivocally synthesized (12). In the present study there seems to be little evidence for crosslinking since the T_g's are so similar.

Chemical Resistance - The chemical resistance of BDSDA/DDSO$_2$ thin film (40 µm thick) to six common solvents was determined and the results listed in Table 4. Methylethyl ketone, xylene, and tricresylphosphate had no visible effect on the film and there was no change in T_g (apparent). Cyclohexanone had only a slight visible effect on the film with no change in T_g. Methylene chloride and cresol caused only minor swelling. The T_g for the methylene chloride soaked film was not measured because the film softened at approximately 110°C preventing a T_g determination. The cresol soaked film exhibited severe shrinkage above 50°C and no T_g was detectable.

SUMMARY

As part of a continuing research program to investigate concepts which will lead to development of high temperature polymers with enhanced hot-melt processability, two similar polyimide systems were synthesized and characterized. The polyimides differed in that the anhydride derived portion contained a sulfide linkage in one and a sulfone linkage in the other. During cure, the sulfide linkage in the first material oxidized to the sulfone, as indicated by infrared analyses. Thus, the slight differences observed in the physical properties of the two materials are probably due to differences in molecular weights.

Both materials exhibited melt-flow. Apparent viscosities were determined using capillary rheology; flow characteristics of both polyimide materials were similar. While both appear to have potential for melt processability with good thermal stability and chemical resistance, they are not as processable as a previous polyimide material (BDSDA/APB) developed earlier in this program.

ACKNOWLEDGEMENTS

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TABLE 1. PHYSICAL PROPERTIES OF POLYMERS

	BDSDA/DDSO$_2$	BDSO$_2$DA/DDSO$_2$
WATER ABSORPTION [a]		
24 hr SOAK	0.25 %	0.25 %
72 hr SOAK	0.43 %	0.42 %
EQUILIBRIUM [b]	0.69 %	0.67 %
DENSITY [c]	1.38 g/cm^3	1.37 g/cm^3
INHERENT VISCOSITY [d]	0.52 dl/g	————
MOLECULAR WEIGHT [e]		
NUMBER AVERAGE ($\overline{M}_n$)	8 500	————
WEIGHT AVERAGE ($\overline{M}_w$)	12 100	————

(a) BAR SAMPLES NOMINALLY 0.254 cm × 0.635 cm × 2.337 cm CUT FROM MOLDED STOCK

(b) EQUILIBRIUM WATER ABSORPTION IN AIR

(c) BDSDA/DDSO$_2$ - MOLDED DISK 5.715 cm DIAMETER × 0.254 cm THICK; BDSO$_2$DA/DDSO$_2$ - BAR SAMPLES NOMINALLY 2.663 cm × 0.639 cm × 2.739 cm CUT FROM MOLDED STOCK

(d) POLYAMIDE-ACID SOLUTION WITH 0.5% SOLIDS IN DMAc AT 35°C

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

TABLE 2. MECHANICAL PROPERTIES OF UNFILLED POLYMERS

	$BDSDA/DDSO_2$	$BDSO_2DA/DDSO_2$
FLEXURAL STRENGTH	27.1 MPa (3.93 ksi)	74.1 MPa (10.7 ksi)
FLEXURAL MODULUS	3.74 GPa (542 ksi)	3.36 GPa (487 ksi)
CRITICAL RATE OF RELEASE OF STRAIN ENERGY, G_{I_c}	268 J/m^2	————

TABLE 3. THERMAL PROPERTIES OF UNFILLED POLYMERS

	BDSDA/DDSO$_2$	BDSO$_2$DA/DDSO$_2$
COEFFICIENT OF THERMAL EXPANSION (30^0C-175^0C)	4.99×10^{-5} $^0C^{-1}$	4.96×10^{-5} $^0C^{-1}$
GLASS TRANSITION TEMP (T_g), CALORIMETRIC	209^0C	205^0C
THERMOMECHANICAL T_g (APPARENT)		
TMA	211^0C	211^0C
DMA	192^0C	200^0C
TBA	208^0C	———
DECOMPOSITION TEMP *	588^0C	575^0C

* 2.5^0C/min HEATING RATE, 15cc/min AIR FLOW, 50% WT. LOSS

TABLE 4. CHEMICAL RESISTANCE OF BDSDA/DDSO$_2$ FILM[a]

SOLVENT	EFFECT	T_g (APPARENT), $^{\circ}$C [b]
METHYLETHYL KETONE	NONE	211 (NO CHANGE)
CYCLOHEXANONE	POSSIBLE SWELLING	213 "
XYLENE	NONE	213 "
TRICRESYLPHOSPHATE	NONE	214 "
METHYLENE CHLORIDE	MINOR SWELLING	NOT DETERMINED [c]
CRESOL	MINOR SWELLING	NOT DETERMINED [d]

(a) 40u THICK FILM

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

(c) FILM SOFTENED AT ~ 110°C AND T_g (APPARENT) COULD NOT BE MEASURED

(d) FILM UNDERWENT SEVERE SHRINKAGE ABOVE 50°C AND T_g (APPARENT) COULD NOT BE MEASURED

WHERE X IS
-S-
OR
-SO$_2$-

DIGLYME/RT

POLYAMIDE-ACID

300^{0}C/SOLID PHASE
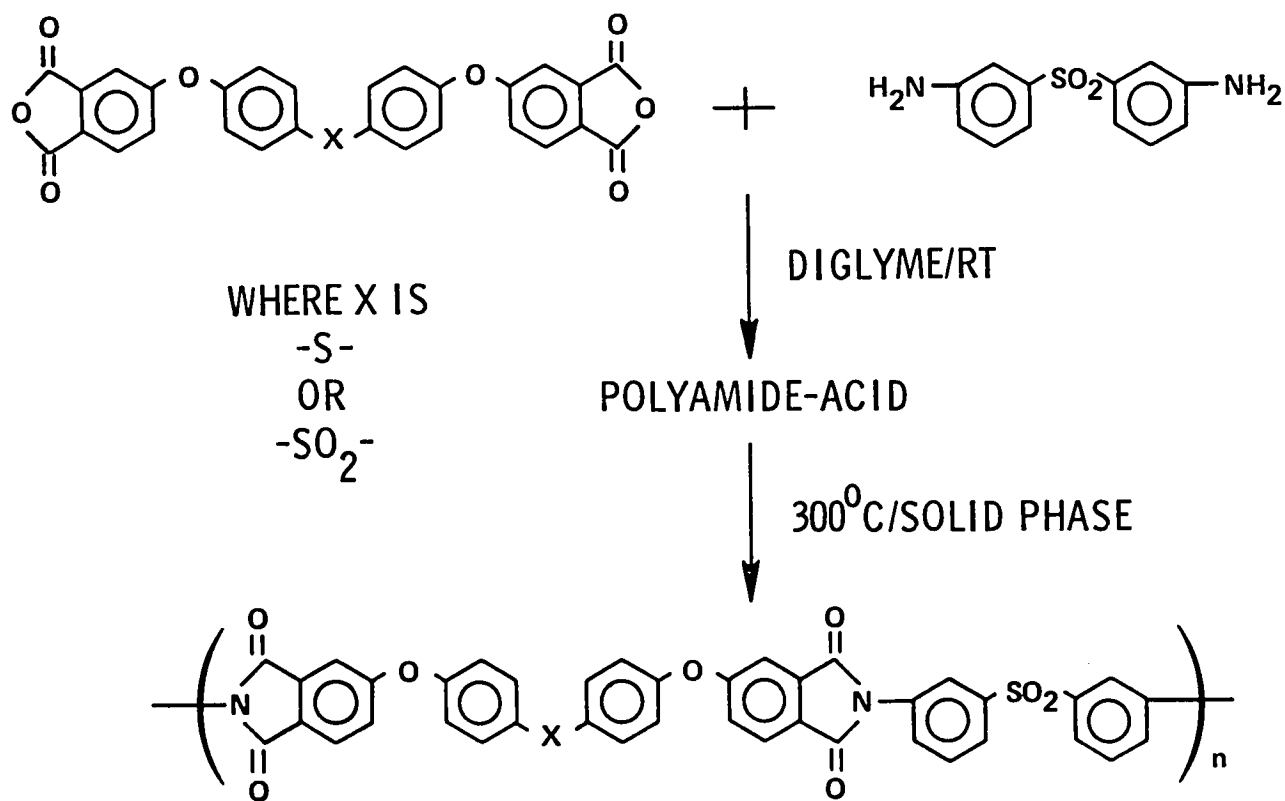
Fig. I.   Polymer Synthesis Scheme For BDSDA/DDSO$_2$ And BDSO$_2$DA/DDSO$_2$.
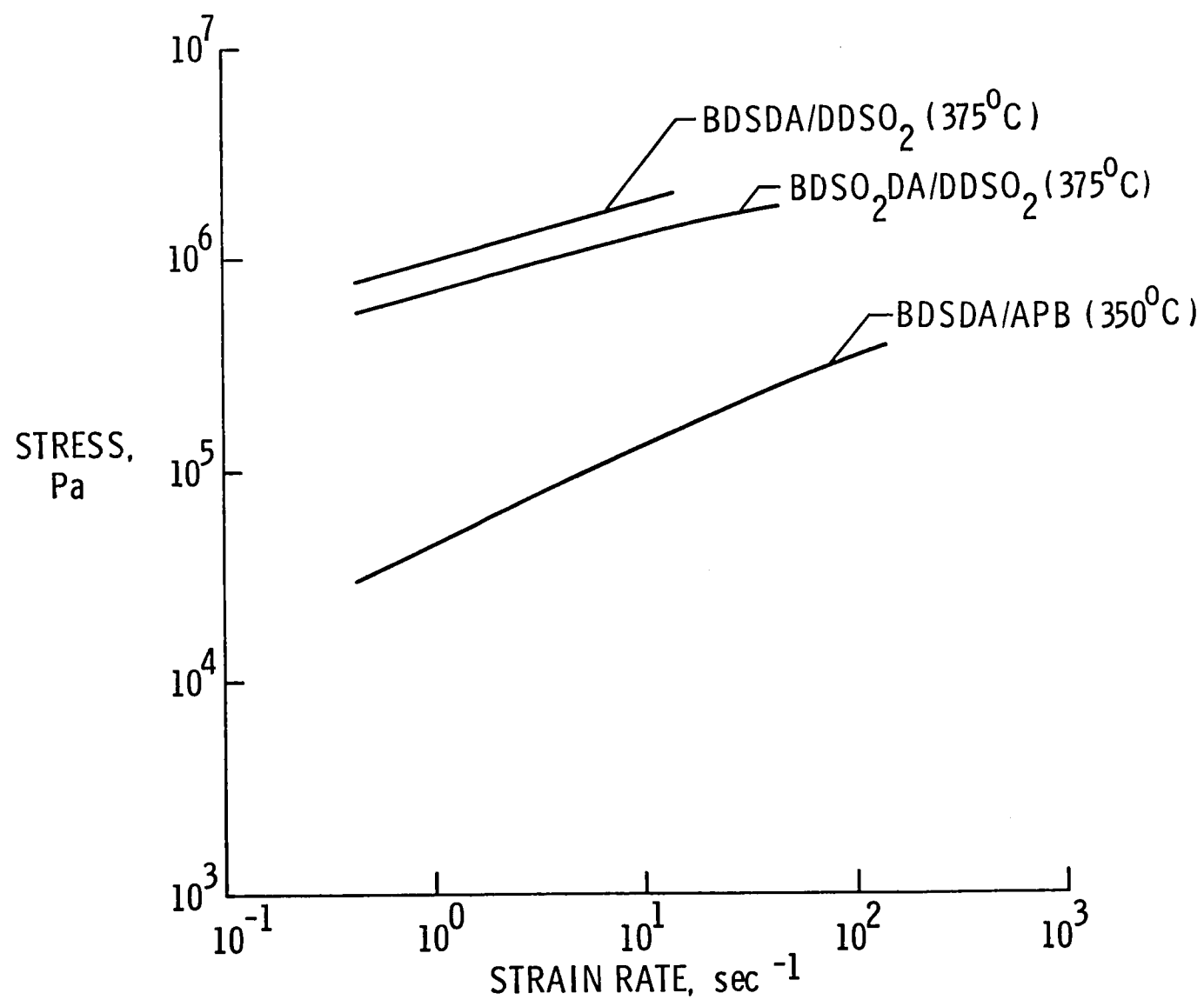
Fig. 2. Capillary Rheometer Stress Vs Strain Rate For BDSDA/DDSO$_2$, BDSO$_2$DA/DDSO$_2$ And BDSDA/APB.
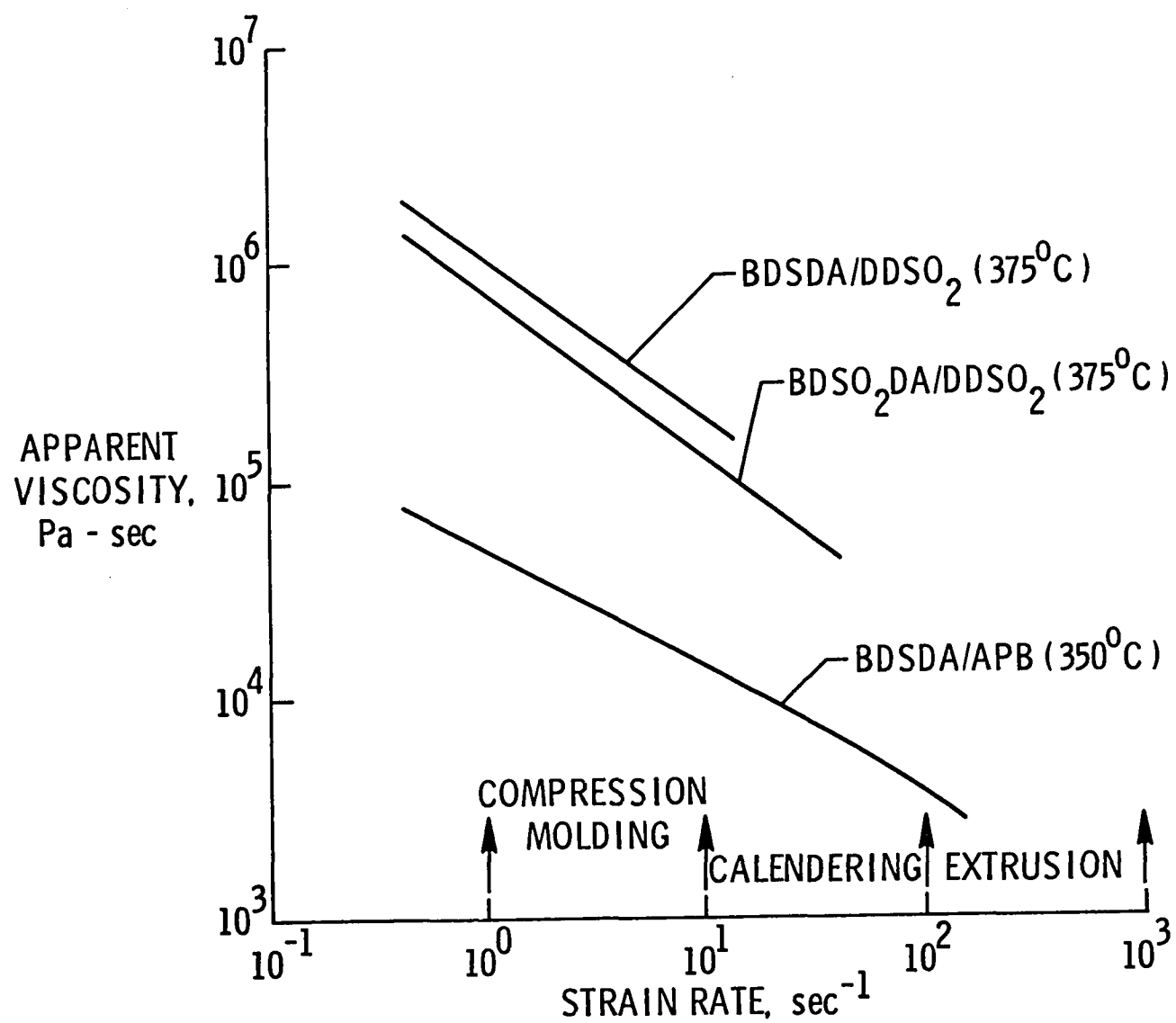
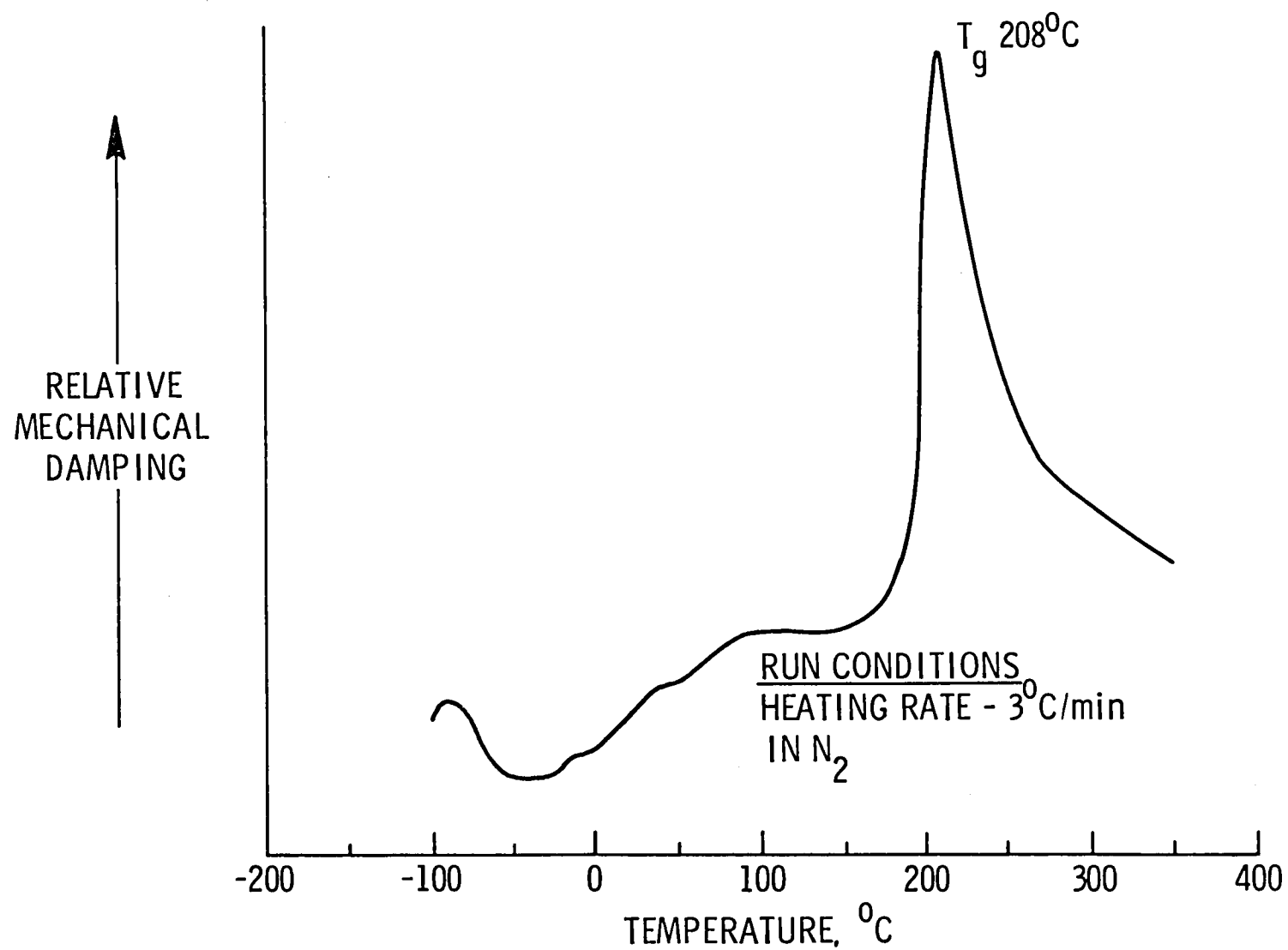Fig. 3. Apparent Viscosity As A Function Of Strain Rate For BDSDA/DDSO$_2$, BDSO$_2$DA/DDSO$_2$ And BDSDA/APB.

Fig. 4. Torsional Braid Analyzer (TBA) Spectrum Of Imidized $BDSDA/DDSO_2$ (Braid precured to $300^\circ C$ in air).
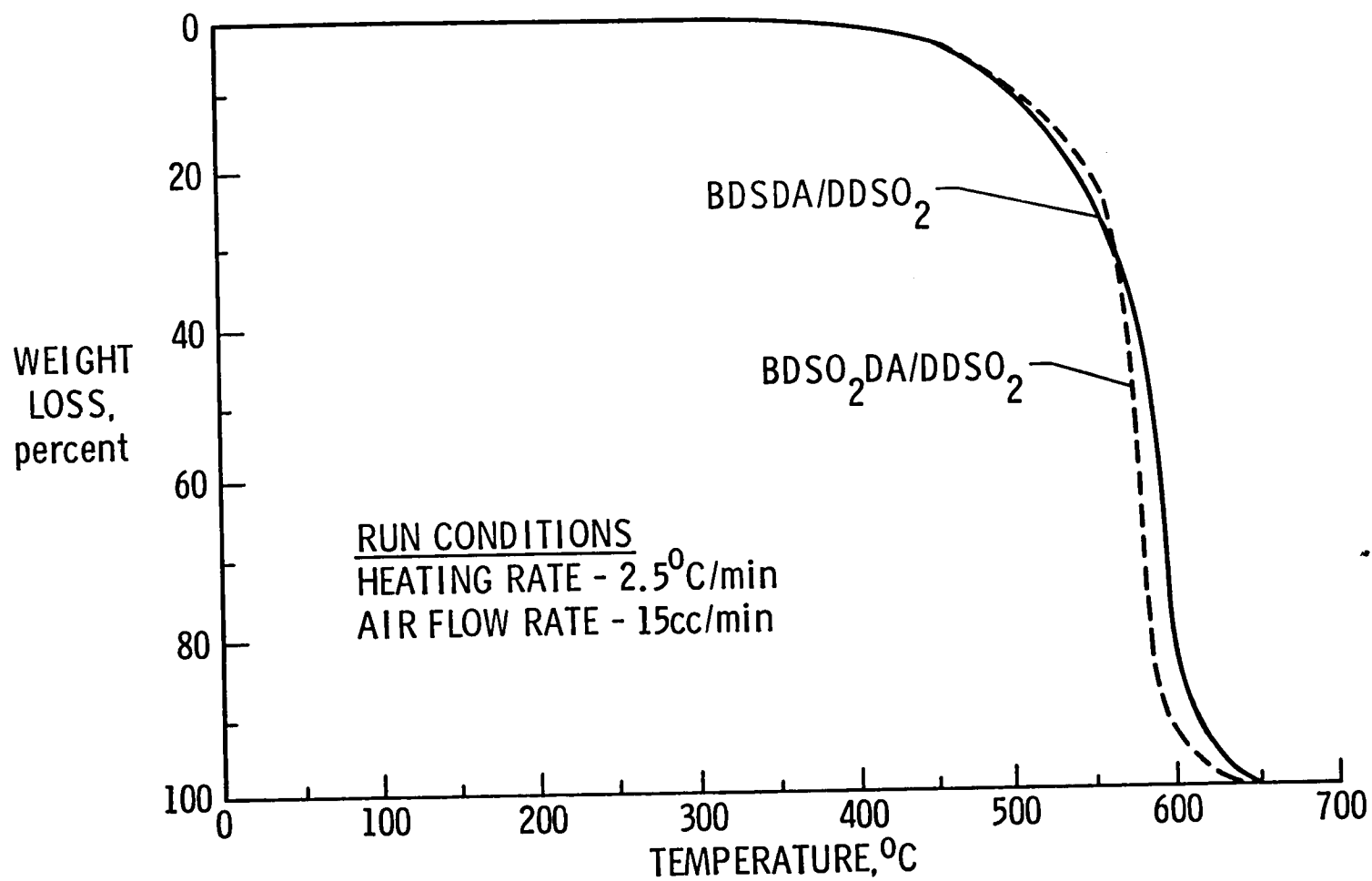
Fig. 5. Comparison Of Thermooxidative Stability Of BDSDA/ DDSO$_2$ And BDSO$_2$DA/ DDSO$_2$ Polymer Film.
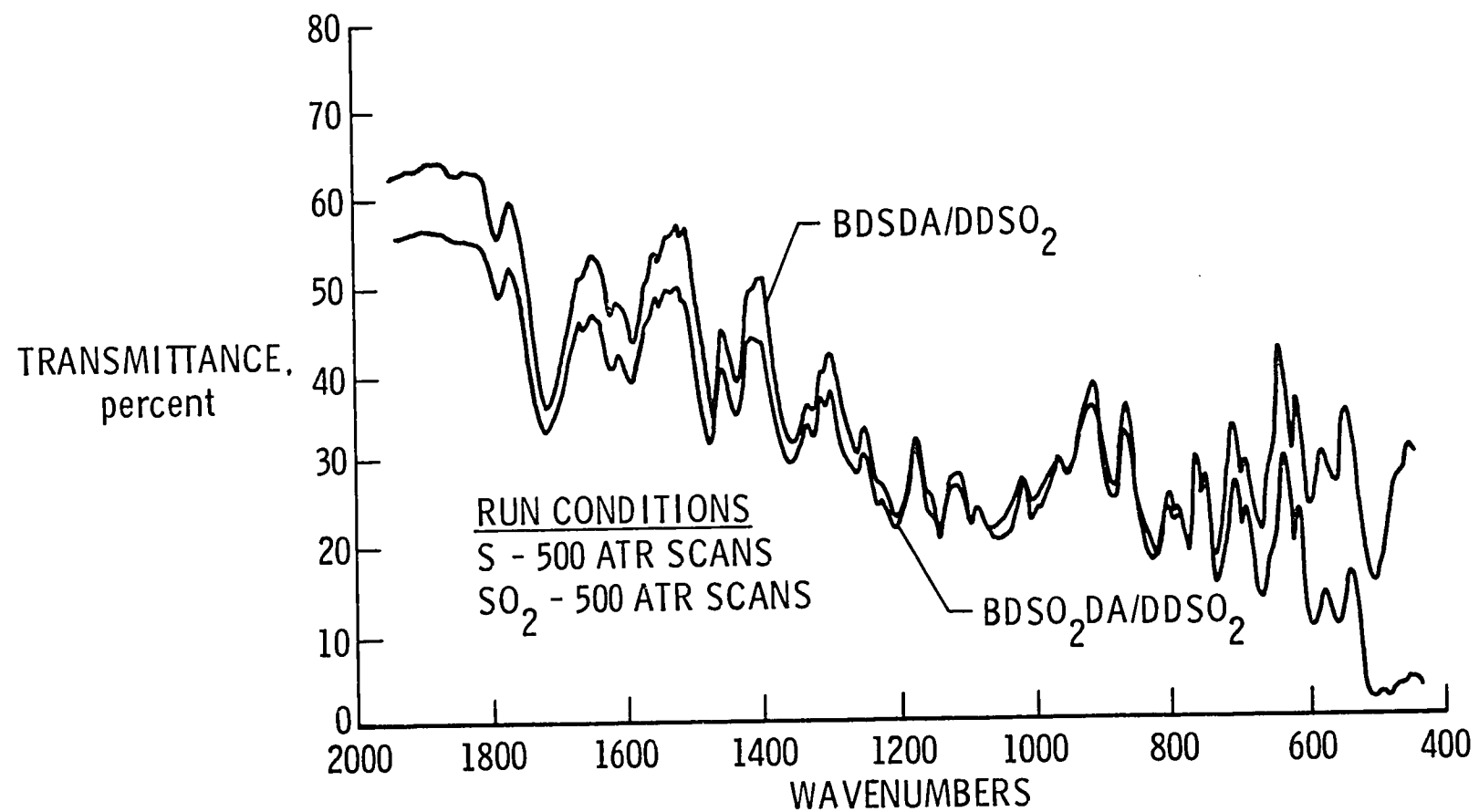
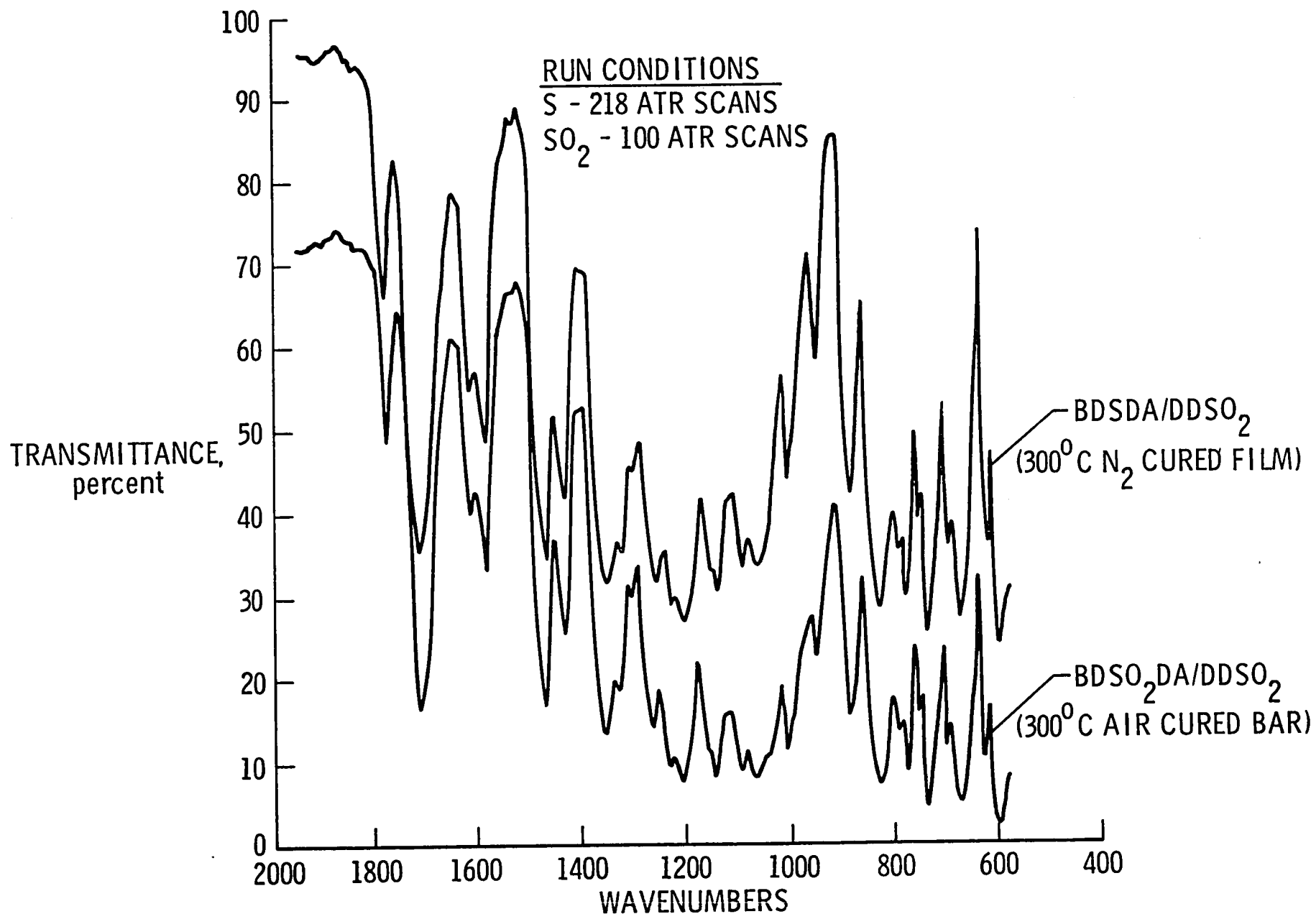Fig. 6. Comparison Of Polymer Moldings By Infrared Analysis.

Fig. 7. Infrared Analysis Of 300°C Air Cured Samples.

1. Report No. NASA TM-84621	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle SYNTHESIS AND CHARACTERIZATION OF A POLYETHERSULFONEIMIDE		5. Report Date April 1983
		6. Performing Organization Code 505-33-33-09
7. Author(s) Harold D. Burks and Terry L. St. Clair		8. Performing Organization Report No.
		10. Work Unit No.
9. Performing Organization Name and Address NASA Langley Research Center Hampton, VA 23665		11. Contract or Grant No.
		13. Type of Report and Period Covered Technical Memorandum
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, DC 20546		14. Sponsoring Agency Code

16. Abstract

Two similar polyimide systems were synthesized and characterized. The only structural difference was a sulfide linkage in the anhydride derived portion of the first system versus a sulfone linkage in the second. Their physical, mechanical, melt-flow and thermal properties were determined and also their resistance to some of the more common solvents. The flow properties of these polyimides indicate them to have potential for melt processability.

17. Key Words (Suggested by Author(s)) Polymer Characterization Flow Properties Polyethersulfoneimide	18. Distribution Statement Unclassified - Unlimited Subject Category 27

19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 26	22. Price* A03