POLYIMIDE FILM " UPILEX"

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Abstract

UPILEX is polyimide film, that the product of the polycondensation reaction between biphenyltetracarboxylic dianhydride(BPDA), of which process Ube Industries, Ltd. (UBE) originally developed, and diamine. Applying BPDA as the acid component, UPILEX of unique properties is obtained. This UPILEX gives useful materials for prominence in heat resistance and other physical and mechanical properties.

Introduction

Polyimides are the most important commercially available high–temperature polymers. They have found many applications in the electronics and aerospace industries due to their excellent electrical, mechanical, and thermal properties. Polyimides are synthesized by the condensation of aromatic dianhydrides and aromatic diamines. Several dianhydrides and diamines have been prepared, but only a few of them are commercially available. UBE has developed a new process to synthesize BPDA on an industrial scale. Applying BPDA as the acid component, polyimides of unique properties are obtained, and the physical properties changes widely according to diamine components. UPILEX–S is polyimide film derived from BPDA with 1,4–diaminobenzene(PPD). UPILEX–S film has proved themselves to be superior in such properties as heat resistance, tensile strength, hydrolysis resistance, dimensional stability and low thermal linear expansion. In this paper, we report properties of the UPILEX–S film.

Experimental

Film preparation.

UPILEX–S films usually are manufactured in a two–step method(Fig.1). The first step is a polycondensation reaction between BPDA and PPD in polar solvents. This polyamic acid solution, which is a polyimide precursor, is then fabricated a film by solvent casting and removal of the solvent. In the second step, the resulting polyamic acid film is converted thermally to the polyimide by the removal of water. UBE has manufactured UPILEX–S films on an industrial scale.

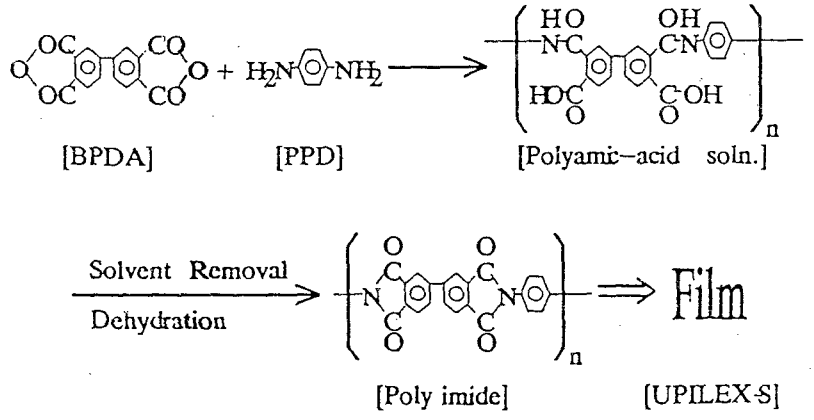

Figure 1. Scheme of two–step polymerization

Analysis

The properties of the UPILEX–S film were obtained according to the ASTM methods.

Results and discussion

Mechanical properties

The characteristics of UPILEX–S film are shown on Table 1 and Figures 2 through 5. Table 1 outlines some of this typical mechanical properties. UPILEX–S film displays superior characteristics not only at ambient temperatures, but at high temperatures as well, with 22Kg/㎟ of tensile strength and 350Kg/㎟ of tensile modulus at 300°C. As shown on Figure 3, UPILEX–S film also features outstanding long–term heat resistance, operating at 290°C(in air) over 20,000 hours with only a 50% reduction in tensile strength. Another advantage inherent in UPILEX–S film is its high resistance to boiling water for long periods of time.

Electrical properties

Table 2 shows some of this typical electrical properties. UPILEX–S exhibits excellent electrical characteristics over a wide range of temperatures and frequencies. Even at high temperatures, UPILEX–S film shows remarkably slight deterioration in its electrical properties.

Thermal properties

The thermal properties of UPILEX–S film are shown on Table 3 and Figures 6 through 7. As shown on these results, UPILEX–S film boasts the highest heat resistance of any plastic film currently available. Its major features include smaller values in both heat shrinkage and thermal linear expansion coefficients, and self–extinguishing (UL94,VTM–0). This makes it ideal for use in FPC(Flexible Printed Circuits) and TAB(Tape Automated Bonding)–tape substrates composed of minute circuits.

Chemical–resistant properties

The chemical resistant properties of UPILEX–S film are shown on Tables 4 through 5. As shown on these results, UPILEX–S film is insoluble in all organic solvents, and is sufficiently resistant to virtually any chemicals, including inorganic acid and alkali solution and so forth. UPILEX–S film features especially high resistance to alkali solutions(NaOH), and retains its physical properties and superior dimensional stability even when exposed to chemicals.

Environmental resistance

UPILEX–S film exhibits superior performance characteristics under a variety of environmental conditions, with advanced features not found in any other polyimide film. Some of these characteristics are low water absorption and superior weather resistance. The characteristics of water absorption are shown on Figures 8 through 10. Generally, polyimide films have one drawback, high water absorption. However, UPILEX–S film features a water absorption value that is half that of conventional polyimide film, and lower absorption/desorption speeds as well. This results in UPILEX–S film exhibiting smaller dimensional changes when exposed to moisture,

changes comparable to polyester, making it perfect for FPC applications where minute circuitry is required.

Another outstanding feature of UPILEX–S film is its high resistance to the weather. Figure 11 displays UPILEX–S film's superior weather resistance characteristics.

Conclusion

As mentioned previously, UPILEX–S film has a well–balanced features on overall characteristics under a wide temperature range. Grades of UPILEX–S film are available ranging from a thin 7.5μm to a thick 125μm, to meet any specific customer application. UBE can also offer extensive consultation services to ensure the satisfaction of customer demands.

References

(1)Ube Industries, Ltd., UPILEX Catalog,1991.

Table 1. Mechanical Properties

Properties	Unit	Typical Values						Test Method
		UPILEX-25S(25μm)				UPILEX-75S(75μm)		
		-269°C	-196°C	25°C	300°C	25°C	200°C	
Tensile Strength (MD)	kg/mm²	57	50	40	22	37	28	ASTM D882
Stress at 5% Elongation (MD)	kg/mm²	—	—	26	9	21	11	ASTM D882
Elongation (MD)	%	7	11	30	48	50	80	ASTM D882
Tensile Modulus (MD)	kg/mm²	—	—	900	350	680	360	ASTM D882
Tear Strength-Initiation [Graves] (MD)	kg/mm	—	—	23	—	30	—	ASTM D1004
Tear Strength-Propagation [Elmendorf] (MD)	g/mm	—	—	330	—	430	—	ASTM D1922
Folding Endurance [MIT]	Cycles	—	—	>100,000	—	>25,000	—	ASTM D2176
Density	g/cm³	—	—	1.47	—	1.47	—	ASTM D1505
Coefficient of Kinetic Friction (film-to-film)	—	—	—	0.4	—	0.4	—	ASTM D1894

*MD: Machine Direction

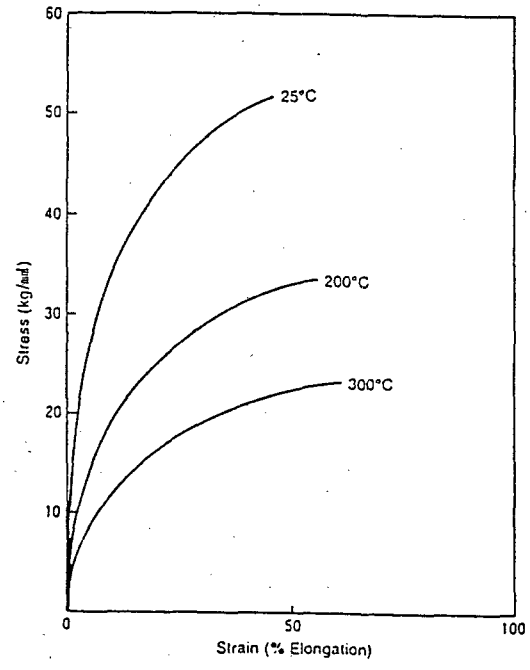

Figure 2. Tensile Strength–Stress Curves

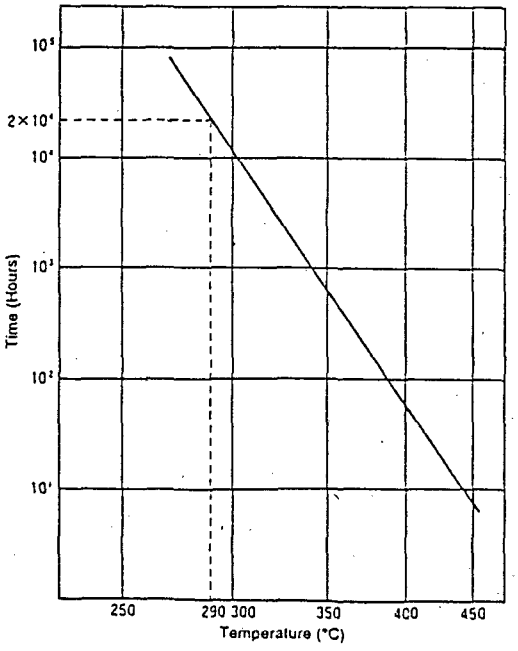

Figure 3. Temperature to 50% Reduction in Tensile Strength

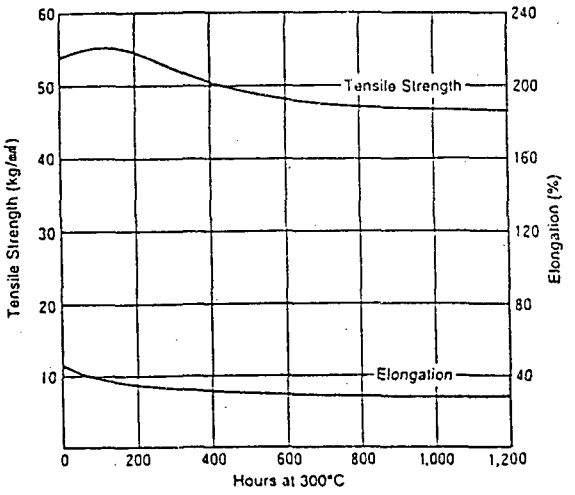

Figure 4. Tensile Strength and Elongation vs. Aging at 300° C

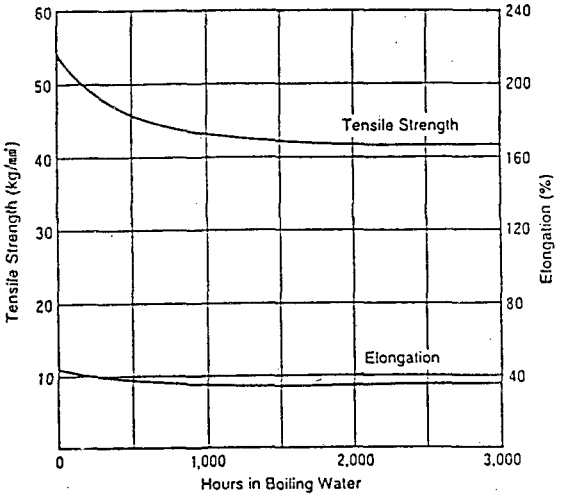

Figure 5. Tensile Strength and Elongation after immersion in Boiling Water

Table 2. Electrical Properties

Properties	Unit	Typical Values				Test Conditions	Test Method
		UPILEX-25S		UPILEX-75S			
		25°C	200°C	25°C	200°C		
Dielectric Strength	kV	6.8	6.8	11	11	50Hz	ASTM D149
Dielectric Constant	—	3.5	3.3	3.3	3.2	10^3Hz	ASTM D150
Dissipation Factor	—	0.0013	0.0078	0.0038	0.0056	10^3Hz	ASTM D150
Volume Resistivity	Ω-cm	10^{17}	10^{15}	10^{16}	10^{14}	DC100V	ASTM D257
Surface Resistivity	Ω	$>10^{17}$	10^{15}	$>10^{16}$	10^{13}	DC100V	ASTM D257

Table 3. Thermal Properties

Properties		Typical Values		Test Conditions (Test Method)
		UPILEX-25S	UPILEX-75S	
Heat Shrinkage (%)		0.2	0.01	200°C, 2Hours JIS C2318
Thermal Linear Expansion Coefficient ($\times 10^{-5}$cm/cm/°C)	20～200°C	1.2	2.0	Values determined by minute linear expansion tester at 5°C/min. temperature increments
Melting Point (°C)		None		
Specific Heat (cal/g/°C)		0.27		Differential Scanning Calorimeter
Temperature Index (°C)		290		Heat Treatment: 20,000Hours
Glass Transition Temperature (°C)		>500		
Flammability		UL94 VTM-0 (Exceeding 7.5μm)		UL94 File No.48133
Oxygen Index (%)		66		JIS K7201
Thermal Conductivity (cal/cm·S·K)		0.04 (50μm)		JIS D1201

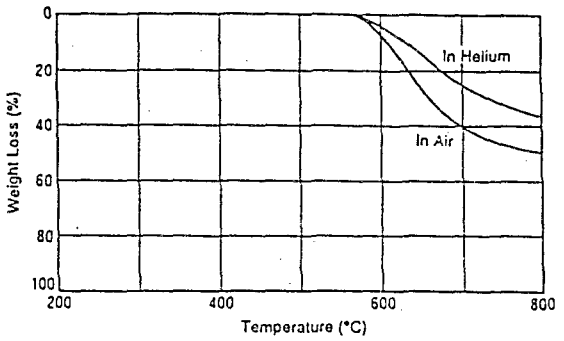

Figure 6. Weight Loss at Temperature Rise in 3° C/min.

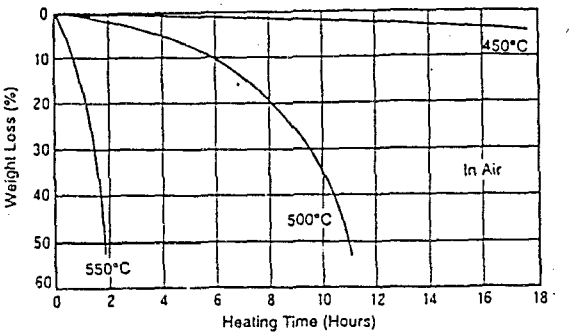

Figure 7. Isothermal Weight Loss

Table 4. Chemical Properties

Properties	Typical Values (25μm Film)(%)			Test Conditions	Test Method
	Strength Retained	Elongation Retained	Modulus Retained		
Resistance to:					
10% Sodium Hydroxide	80	60	95	Immersion at 25°C for 5 days	ASTM D882
Glatial Acetic Acid	100	95	100	Immersion at 110°C for 5 weeks	
Water PH=1.0	95	85	100	Immersion at 100°C for 2 weeks	
PH=4.2	95	85	100	Immersion at 100°C for 2 weeks	
PH=8.9	95	85	100	Immersion at 100°C for 2 weeks	
PH=10.0	95	85	100	Immersion at 100°C for 4 days	
Water Absorption	1.4%			Immersion in water at 23°C for 24 hours	ASTM D570
	0.8%			Equilibrium at 60%RH, 50°C	
Gas Permeability					
Water Vapor	1.7g/m²/mil			At 38°C, 90%RH for 24 Hours	ASTM E96
Oxygen	0.8ml/m²/mil			At 30°C, 1 atm for 24 Hours	ASTM D1434
Carbon Dioxide	1.2ml/m²/mil			At 30°C, 1 atm for 24 Hours	ASTM D1434

Table 5. Dimensional Stability When Immersed in Various Chemical Solutions and Solvents

(Unit: %)

Chemicals	Typical Values (25μm Film)		Immersion Conditions
	MD	TD	
Ferric Chloride (37%)	−0.01	+0.01	At room Temperature for 10 minutes
Cupric Chloride (37%)	+0.01	−0.01	At room Temperature for 10 minutes
5% Sodium Hydroxide	−0.02	+0.03	At 60°C for 30 minutes
Isopropanol	−0.00	+0.01	At room Temperature for 10 minutes
Methyl Ethyl Ketone	−0.01	−0.00	At room Temperature for 10 minutes
Methylene Chloride/Trichloroethane (Mixed)	−0.00	+0.00	At room Temperature for 10 minutes
2N-Hydrochloric Acid	−0.00	−0.00	At room Temperature for 10 minutes

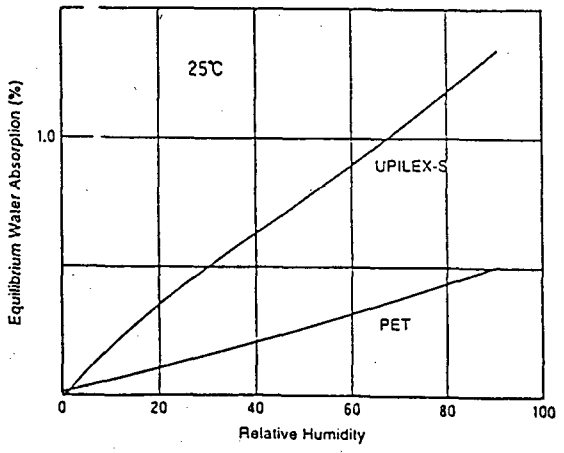

Figure 8. Equilibrium Water Absorption vs. Relative Humidity

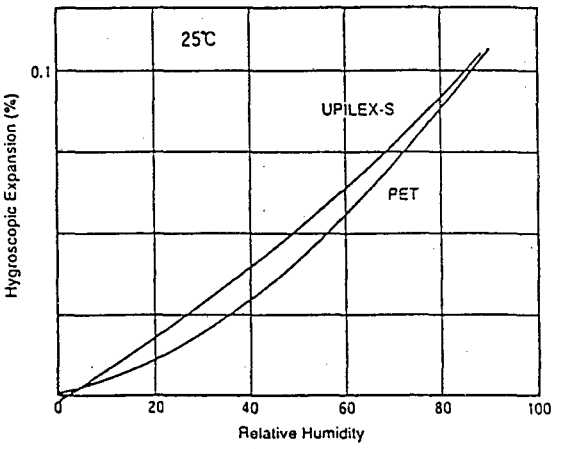

Figure 9. Hygroscopic Expansion vs. Relative Humidity

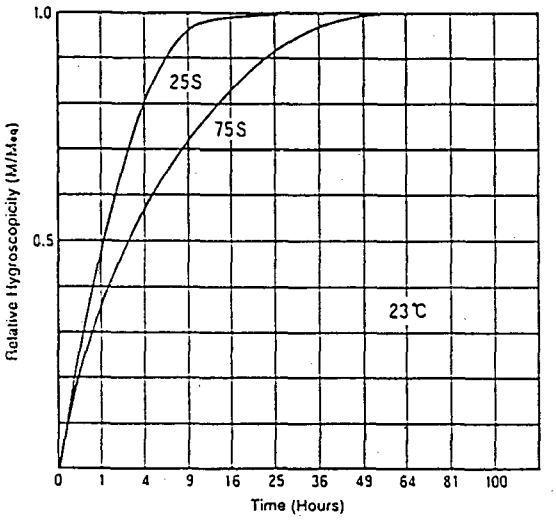

Figure 10. Hygroscopicity

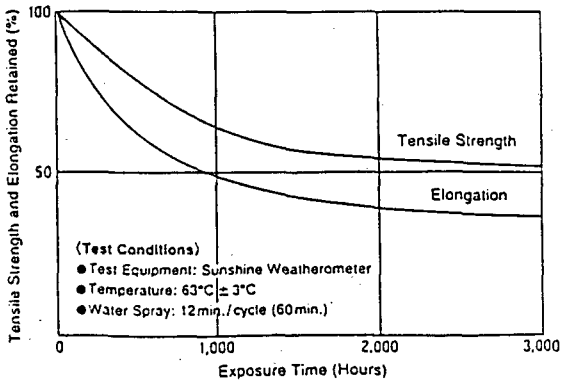

Figure 11. Weather Resistance