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Polyimide in Electronics: Applications and Processability Overview

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Abstract

Polyimides are nowadays quite famous dielectrics and insulating materials widely used in electronics and electrical engineering applications from low voltage microelectronics up to high voltage engineering industry. They are well appreciated because of their excellent physical properties (i.e., thermal, electrical, and mechanical properties), as well as, their coating process ease either from a liquid or a gas phase. Consequently, polyimides appear in a various range of applications to efficiently separate metal levels or electrodes at different electrical potentials. This chapter intends to review the main chemical generalities of polyimides, the different monomer families, the coating and curing processes, and the main physical properties for electronic and high voltage industrial applications.

Keywords: polyimide, imidization, deposition techniques, curing process, physical properties, microelectronics, high voltage

1. Introduction

Polyimides are advanced polymeric materials that are well known for their excellent thermal, electrical, mechanical, and chemical properties [1]. Polyimides are particularly interesting for microelectronics as well as for high voltage electrical engineering industry, thanks to their high thermal stability, high glass transition temperature, low dielectric constant, and overall very good electrical insulation properties (high breakdown field, low dielectric loss factor, and low conductivity). Polyimides present also an ease of processability making them patternable for many types of integrated electronic devices [2]. In the last 20 years, with the emergence of a wide range of novel electronic applications, polyimides have regained an increasing interest from both fundamental researches and applicative research and development sides, as shown in **Figure 1** where the publication map across the scientific fields and the total number of publications in Electrical Engineering and Electronics edited journals between 1975 and 2019 are presented.

Thus, one can observe that two publication thresholds occurred in 1999 and 2011 leading now to an annual dissemination activity on polyimides close to 4,000 publications for only the Electrical Engineering and Electronics domains. This clearly proves the large interest that these polymeric materials arouse and this trend should still progress over the next coming years.

Polyimides are present in different ways (from substrates to thin coatings) and for different purposes (purely mechanical one to advanced electrical insulation) in

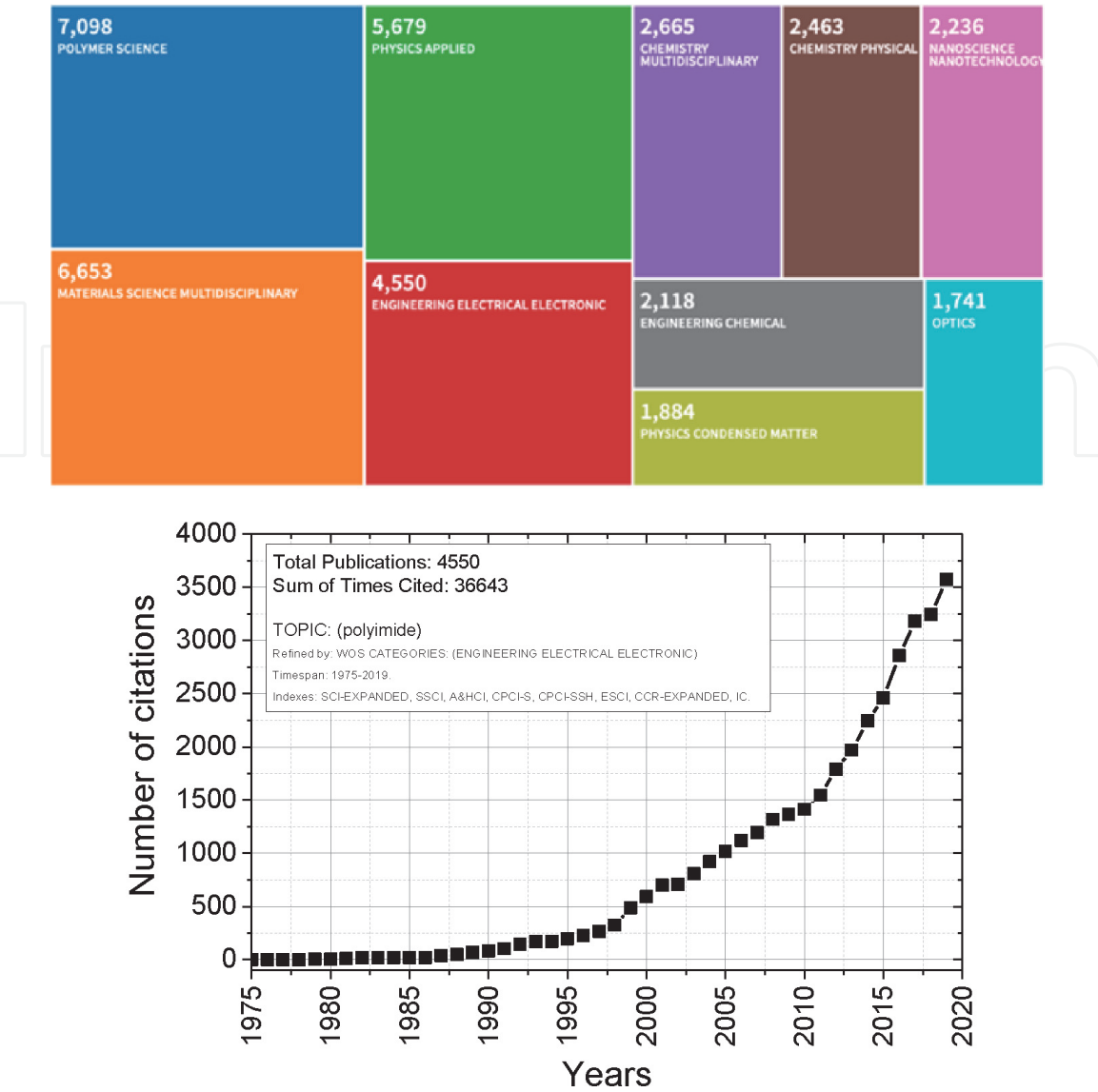


Figure 1. All publication records map on 'polyimide' ranked by topics from 1975 to 2019 (a) and number of citations per year in the category 'Electrical Engineering Electronic' (source: Web of science).

electronics and high voltage applications. **Figure 2** shows an overview of the main electrical industrial applications of polyimides.

They can be listed as:

- low- k intermetallic layer for ultra-large scale integration in microelectronics,
- bond pad redistribution and buffer layers for ICs
- films for flexible substrate for the development of a wide range of sensors,
- rigid substrate for PCB technologies,
- protective coating for space applications,
- films for high temperature capacitors in new energy storage challenges,
- insulating layer in next generation of high temperature aeronautic cables,

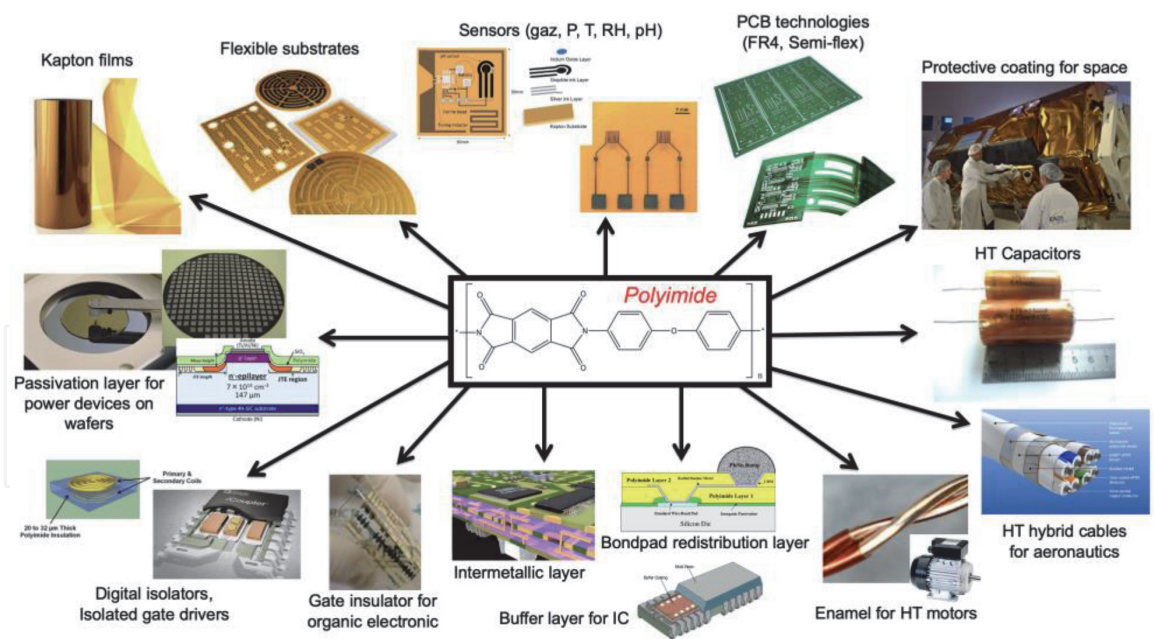


Figure 2.
 Overview of the main electronic and HV engineering industrial applications of polyimide.

- high thermal class enamel for high temperature wires in electrical motor applications,
- passivation layer for surface insulation of high voltage and/or high temperature power electronic devices (Si, SiC, GaN, ...),
- insulating barrier for digital isolators in isolated gate drivers,
- gate insulator for new organic electronics, etc.

The present book overall intends to present most of these applications and the place of polyimides in their related context, the state-of-the-art as well as the next challenges to face to enable improving the efficiency of electrical devices and systems at different scales.

As a preliminary introduction, the present chapter will detail some generalities on polyimide chemistry, their thermal stability, the main chemical routes to synthesize them, their deposition and process techniques, their different curing processes, and a summary of their main physical properties.

2. Chemical generalities of polyimide

2.1 Thermal stability

The thermal stability of a polymer is defined as its ability to withstand high temperatures without initiating degradation processes such as thermolysis. It is usually evaluated by thermogravimetric analysis. This method consists in measuring the mass loss of a material either as a function of temperature or as a function of time at a constant temperature. By convention, a polymer is said to be thermostable if it can be used without losing its properties for 1000 hours at 300°C, 10 hours at 400°C, and a few minutes at 500°C. In the case of polyimides, the decomposition temperature generally appears between 500°C and 600°C. However, their typical

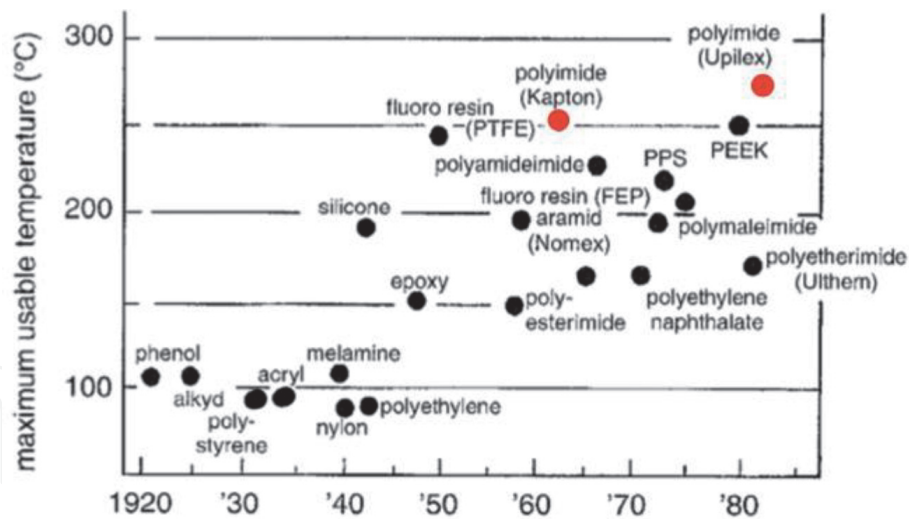


Figure 3.

Maximum working temperature for the main developed polymers over the last century including polyimides (reproduced and modified from [1]).

maximum usable temperature is usually between 250°C and 275°C, as shown in **Figure 3** where all the main thermostable polymers are also displayed for comparison. Thus, polyimides appear as the thermostable polymer having the highest maximum working temperature.

It has been shown that the increase in the number of benzene rings in polyimide monomer macromolecules contributes to increase their degradation temperature [1]. However, the degradation temperature can also be affected by the presence of low thermostable chemical bonds in the macromolecular structure like the C—O—C ether group [3]. Finally, a few studies even present thermal stability as high as 300°C that report on the potential use of polyimides in high temperature electronic applications for electrical insulation purposes [4, 5].

2.2 Synthesis and imidization reaction

The two-step synthesis method is the simplest and most commonly used method to obtain polyimides in industry. In 1955, Edwards et al. were the first to synthesize polyimides (PI) from polyamide salts [6]. Endrey was the first to successfully synthesize high molecular weight aromatic polyimides [7]. In the method described, the synthesis is carried out in two stages.

The first one is to prepare a polyamic acid (PAA) solution, which is the precursor of polyimide. The synthesis of PAA takes place via the reaction between two precursor monomers, a dianhydride and a diamine at room temperature and in polar aprotic solvents such as N-methyl-2-pyrrolidone (NMP), N,N-dimethylformamid (DMF) or N,N-dimethylacetamid (DMAc).

PAA is then cyclodehydrated using a thermal or chemical conversion process, called “imidization,” to form the final, insoluble, and infusible polyimide. The steps for the synthesis and imidization of polyimides by this method are presented in **Figure 4**, where —R— and —R’— represent the radicals of the dianhydride and diamine monomers, respectively.

2.3 Main radicals for dianhydride and diamine precursor monomers

Many varieties of PAA can be synthesized leading to hundreds of different polyimide combinations. **Tables 1** and **2** list the main radicals —R— and —R’— of the dianhydride and diamine monomers, respectively, marketed for the synthesis of PAA.

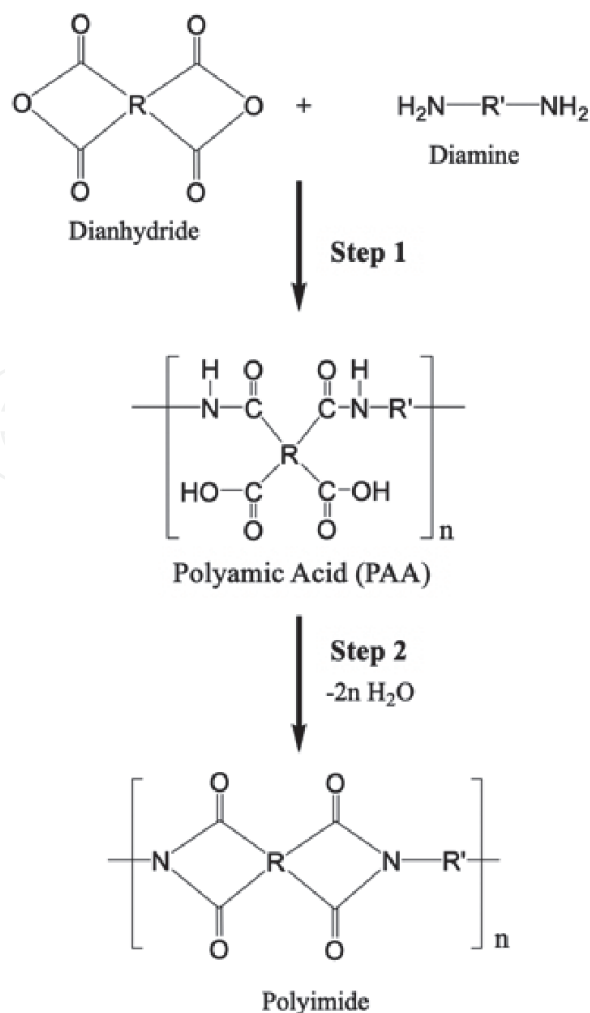


Figure 4.
 Two-steps synthesis method of polyimide. Step 1: PAA synthesis. Step 2: PAA conversion into polyimide by imidization.

2.4 Polyimide deposition and process techniques

There are several routes to shape polyimides depending on the targeted application. Among them, one can cite the biaxial stretching for film production, the spin-coating and lithography process for wafer-level deposition and the vapor-deposition process. The following sections present all these.

2.4.1 Biaxial stretching for film production

Compared with the lab-scale preparation, the greatest difference for industrial manufacturing of polyimide films is the stretching process [9, 10]. Stretching process, either uniaxial or biaxial stretching of the gel-like PAA films, will result in the full orientation and extension for the polyimide molecular chains.

Figure 5 shows the main steps of the industrial scale production line of polyimide film by biaxial stretching from PAA precursors. In this procedure, the precursor monomers are firstly introduced into the polymerization reactor containing the solvent. After PAA synthesis, the obtained solution is deaerated and cast in the form of a continuous film onto the surface of a heated rotating stainless steel drum. The solvent is partially evaporated and a part of the imidization reaction takes place simultaneously. Thus, a self-supported PAA film is formed. The gel-like PAA film is peeled from the metal drum and first stretched in the machine direction (MD) while controlling the stretching rate. The PAA film is then stretched in the transverse direction (TD). The solvent is removed by evaporation, and the film is heat treated by means of hot air or radiant heat from an


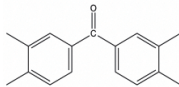
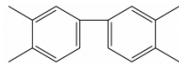
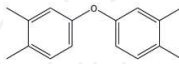
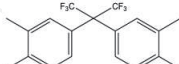
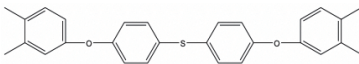
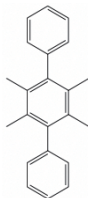
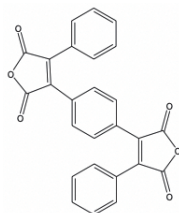
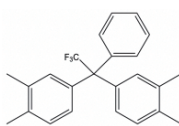
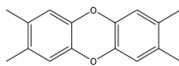
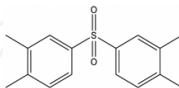
Dianhydride		Radical chemical structure (—R—)
Pyromellitic dianhydride	PMDA	
3,3',4,4'-benzophenonetetracarboxylic dianhydride	BTDA	
3,3',4,4'-biphenyltetracarboxylic dianhydride	BPDA	
3,3',4,4'-oxydiphthalic anhydride	ODPA	
4,4'-hexafluoroisopropylidenebis(phthalic anhydride)	6FDA	
4,4'-bis(3,4-dicarboxyphenoxy) diphenylsulfide dianhydride	BDSDA	
3,6-diphenylpyromellitic dianhydride	DPPMDA	
1,4-phenylenebis-(phenylmaleic anhydride)	1,4-P(PMA)	
4,4'-(1-phenyl-2,2,2-trifluoroethylidene bis (phthalic anhydride)	3FDA	
4,4',5,5'-dioxydiphthalic anhydride	DODPA	
4,4',5,5'-sulfonyldiphthalic anhydride	DSO ₂ DA or DSDA	

Table 1.
Main radicals for dianhydrides (reproduced from [8]).

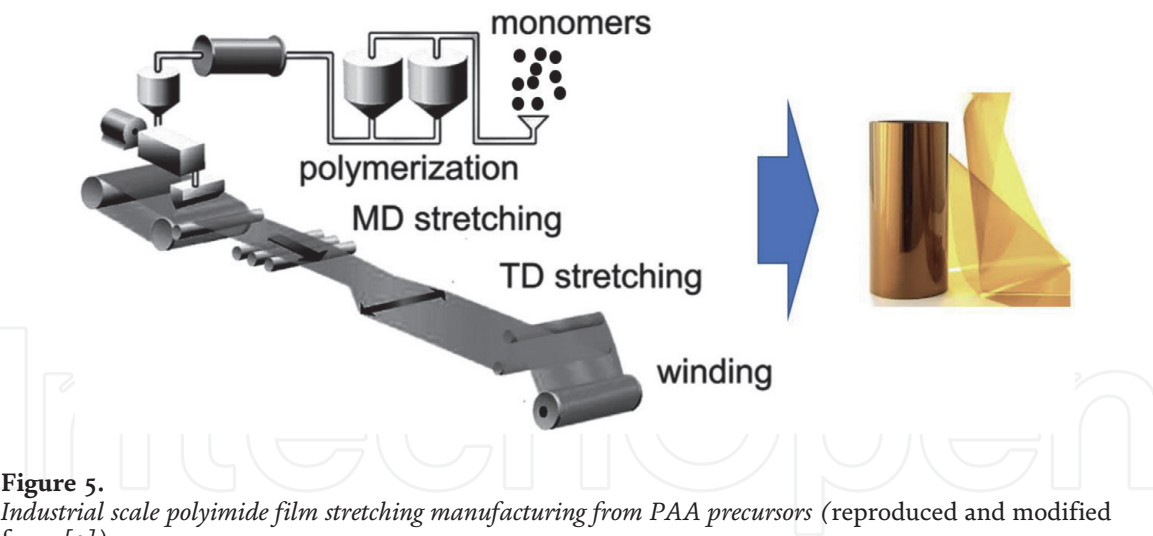
electrical heater to give a biaxial oriented polyimide film. The transverse stretching is carried out at temperatures around 350°C to facilitate the imidization of PAA into polyimide. Such a procedure has been widely used for PI film production, and there has been significant patent activity in the past half century since the commercialization of PI films in 1960s. Up to now, most of the commercially available wholly aromatic PI films have been produced by such kind of procedure.

2.4.2 Spin coating and lithography for wafer-level deposition

Spin coating is a common technique widely used in microelectronic industry for applying resins in thin film form to silicon wafers. Its primary advantage over other methods is its ability to quickly and easily produce very uniform films.

Diamine		Radical chemical structure (—R'—)
<i>p</i> -phenylene diamine	<i>p</i> -PDA, PPD	
<i>m</i> -phenylene diamine	<i>m</i> -PDA, MPD	
4,4'-methylene dianiline ou 4,4'-diaminodiphenyl methylene	MDA	
2-chlorophenylene diamine	C1PPD	—
benzidine	Bz or BDA	
2,2'-dichlorobenzidine	2,2'-diC1Bz	—
3,3'-dimethylbenzidine	3,3'-diMeBz or DBDA	
4,4'-oxydiphenylene ether or 4,4'-diaminodiphenyl ether	ODA or DDE	
4,4'-diaminobenzophenone	4,4'-DABP	
3,3'-diaminobenzophenone	3,3'-DABP	
4,4'-diaminodiphenyl sulfone	4,4'-SO2D or DDS	
4,4'-diaminodiphenyl sulfide	SDA	
1,4-bis-(4-aminophenoxy)benzene	APB 4-1,4	
1,4-bis-(3-aminophenoxy)benzene	APB 3-1,4	—
1,3-bis-(4-aminophenoxy)benzene	APB 4-1,3	
1,4-phenylindanc diamine	DAPI	
2,2-bis-[4-(4'-aminophenoxyphenyl)] hexafluoropropane	4-BDAF	
1,1-bis-(4-aminophenyl-1-phenyl-2,2,2) trifluoroethane	3FDAM	
2,2-bis-(4-aminophenyl) hexafluoropropane	<i>p,p'</i> -6FDAM	
2,2'-di-bis-(trifluoromethyl) benzidine	PFMB, TFMB	
3,5-diaminobenzotrifluoride	DABTF	

Table 2.
Main radicals for diamines (reproduced from [8]).



When the solution of PAA containing NMP solvent is spun at high speeds, the centrifugal force enables to cover the substrate (see **Figure 6**). Spin coating results in a PAA thin film ranging from a few microns to a few tens of microns in thickness depending on coating parameters. The thickness of the final film is determined by the spinning speed, surface tension, and viscosity of the solution. The solvent is removed partly during the spinning process due to evaporation and partly by subsequent baking at elevated temperatures during imidization.

Many experiments have confirmed a mathematical model for photoresist deposition where the thickness variation versus the spin speed is like $\omega^{-0.5}$ [11]. In the

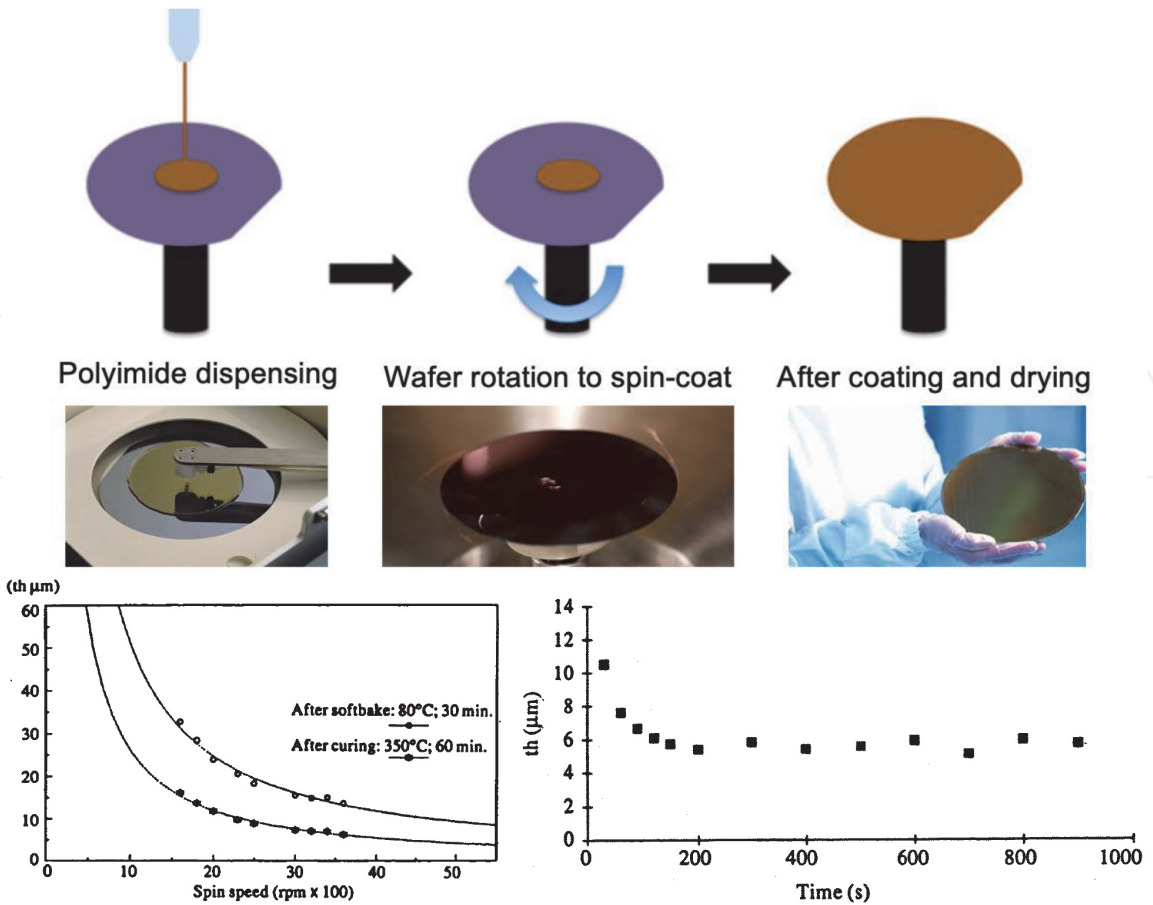


Figure 6. (a) Industrial polyimide spin-coating process on 8''-wafer scale from PAA precursor solution. (b) Polyimide thickness as a function of spin speed and time (reproduced from [11]).

case of polyimide deposition, the thickness variation versus the spin speed shows experimentally different behavior ranging between $\omega^{-0.5}$ and ω^{-1} . It has been proved that the polyimide has a different behavior depending on the residual solvents at the end of the process. The double behavior is emphasized with two different power laws. This proves that the model is verified only if the solvent is completely removed at the end of the process.

Nowadays, the semiconductor manufacturing industry commonly process 300 mm wafers. Thus, polyimides have been developed to be spin-coated and patterned at this scale with very good thickness uniformity accuracy (around 2% of difference in thickness across the wafer) [12].

Conventional, or non-photosensitive, polyimide cannot be directly patterned on wafers due to the absence of photo-active agent (see **Figure 7a**). They require several process steps after the fabrication of the active device. To process non-photosensitive polyimide, a first thick polyimide film is spin coated on the wafer similarly to the photoresist process (see **Figure 7b**). Then, a thin layer of photoresist is applied and exposed using a photolithography tool. A standard development process of the photoresist using a mask is used to define the pattern. This pattern is transferred to the polyimide layer by wet etching through openings during the photoresist lithography step. The wet etch is an isotropic process that causes critical dimension and sidewall control issues. This technical difficulty, combined with the complexity of the process, has limited the non-photosensitive polyimide application.

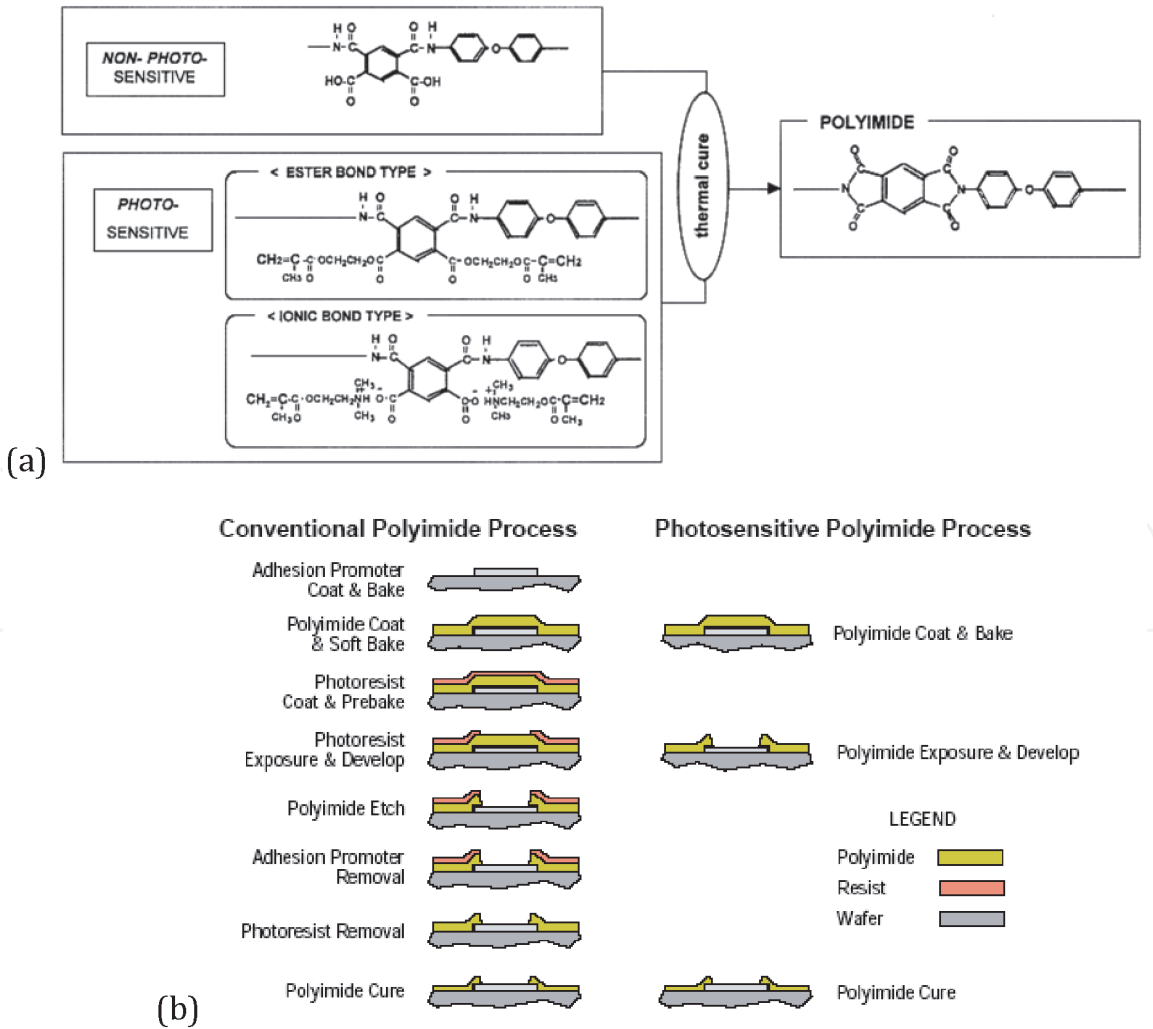


Figure 7.
Comparison of precursor PAA monomers between non-photosensitive and photosensitive polyimides (a).
Lithography process steps comparison between conventional and photosensitive polyimides (reproduced from [13]) (b).

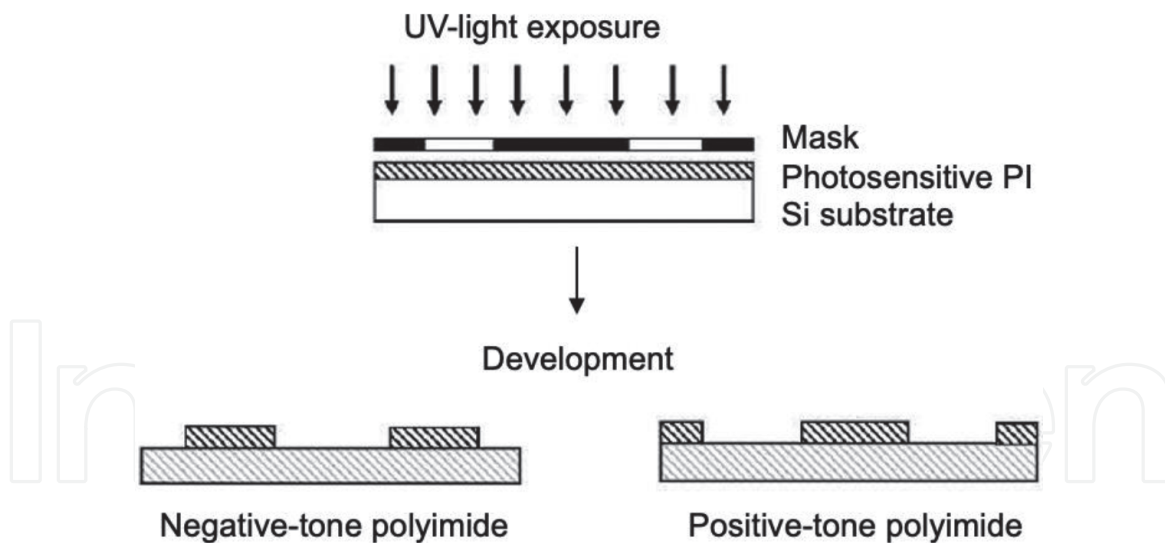


Figure 8.
Lithography process steps comparison between negative and positive photosensitive polyimide resins.

To overcome such difficulties, photosensitive polyimides have been developed to offer an alternative cost savings to the buffer coat polyimide application (see **Figure 7a**). Photosensitive polyimides can be processed similarly to standard resists using photolithography techniques, as shown in **Figure 7b**. Thus, the eight-step non-photosensitive polyimide process can be reduced into a three-step process using photosensitive polyimide. In addition to providing process simplification, this three-step process offers the significant advantages of superior resolution and improved sidewall profiles. As a consequence, cycle time and chemical consumption are reduced. All of these benefits translate into cost savings, ease of use and better quality.

Photosensitive polyimide, like photoresist, can be divided into two categories: positive and negative tones (see **Figure 8**). In the case of the positive tone, the photosensitive polyimide is degraded by UV light and the developer will dissolve away the regions that were exposed. That will leave behind the coating where the mask was initially placed. In the case of the negative tone, the photosensitive polyimide is cross-linked by UV light and the developer will remove only the unexposed regions, leaving behind the coating in areas where the mask was not placed. Application of positive photosensitive polyimide is limited because of the narrow film thickness range available. This makes the negative photosensitive one the most commonly used in electronic industry with a wide viscosity range.

2.4.3 Vapor-deposition process (VDP)

The vapor-deposition polymerization (VDP) is a method where polyimide is directly deposited and synthesized from its two precursor monomers (dianhydride and diamine), evaporated separately at high temperature in a vacuum chamber and collected on a heated substrate for imidization (see **Figure 9**).

The temperature of the transported vapors and of the substrate is usually between 100 and 200°C. A higher temperature post-annealing ($\geq 300^\circ\text{C}$) is sometimes carried out to complete the imidization reaction. The synthesis of polyimides (with thickness between a few 1 to 10 μm) has been successfully demonstrated by this method for PMDA/ODA [16–19] and other variants of polyimides. Polyimides synthesized by VDP generally have a low oxygen permeability in comparison with conventional spin-coated versions and adhere relatively well to their substrate. However, this method remains difficult to fit with industrial manufacturing processes for electrical and electronic systems because of certain inhomogeneities in terms of thickness of the layers.

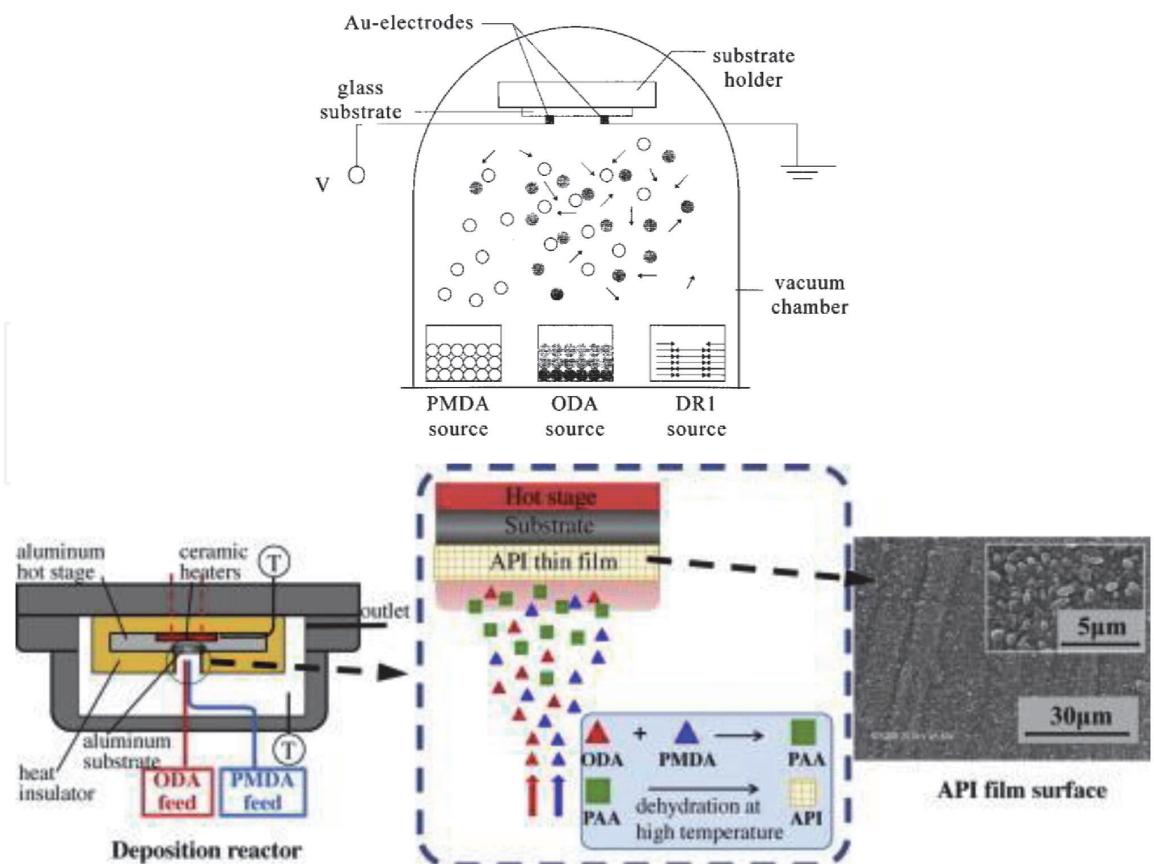


Figure 9.
 Polyimide thin-films deposited by VDP from vapor phase (reproduced from [14, 15]).

2.5 Curing process techniques

2.5.1 Thermal curing

Different techniques for curing polyimides and complete the imidization reaction can be used and are reported in the literature. The curing enables to convert PAA into polyimide and so that to finalize the physical properties of the deposited layer. The most commonly used method is the thermal curing, which is carried out optimally at temperatures of at least 250°C under inert gas, like N₂. It is simple to implement and leads to good properties of the material.

During that critical process step, the PAA coating is slowly heated up until a first temperature plateau at 200°C corresponding to the NMP solvent boiling point. It is usually held on for at least 15 minutes in order to fully remove the solvent from the layer (see **Figure 10**). Then, a subsequent temperature rising is performed up to a second plateau at temperature from 250 to 400°C and between 30 minutes up to 2 hours to complete the imidization reaction and obtain the final polyimide. Of course, the final chemical structuration and physical properties strongly depend on the temperature and time duration [20]. Moreover, heating and cooling ramps also need to be controlled to avoid thermomechanical stress storage within the films [21].

2.5.2 Variable frequency microwave (VFM) curing

In addition, other annealing methods have started to emerge, leading to equivalent properties of the deposited polyimide layers. This is the case with variable frequency microwave (VFM) curing [22, 23].

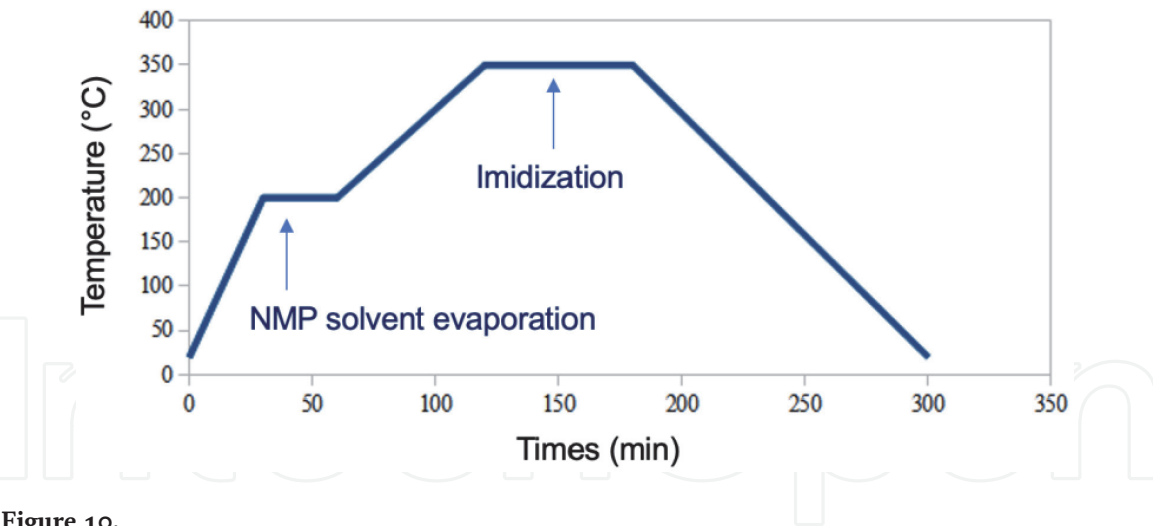


Figure 10.
Typical thermal curing profile for polyimide.

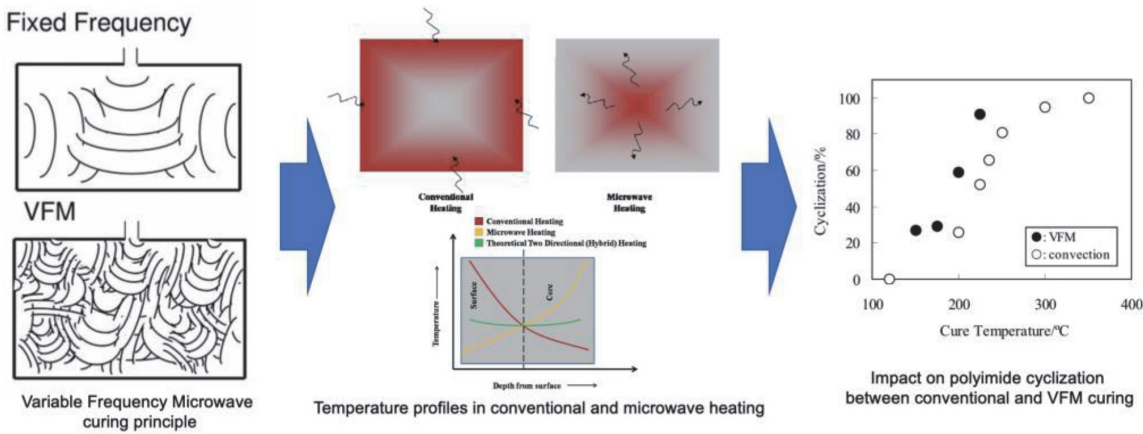


Figure 11.
Variable frequency microwave curing process for polyimide and impact on cyclization (reproduced from [25]).

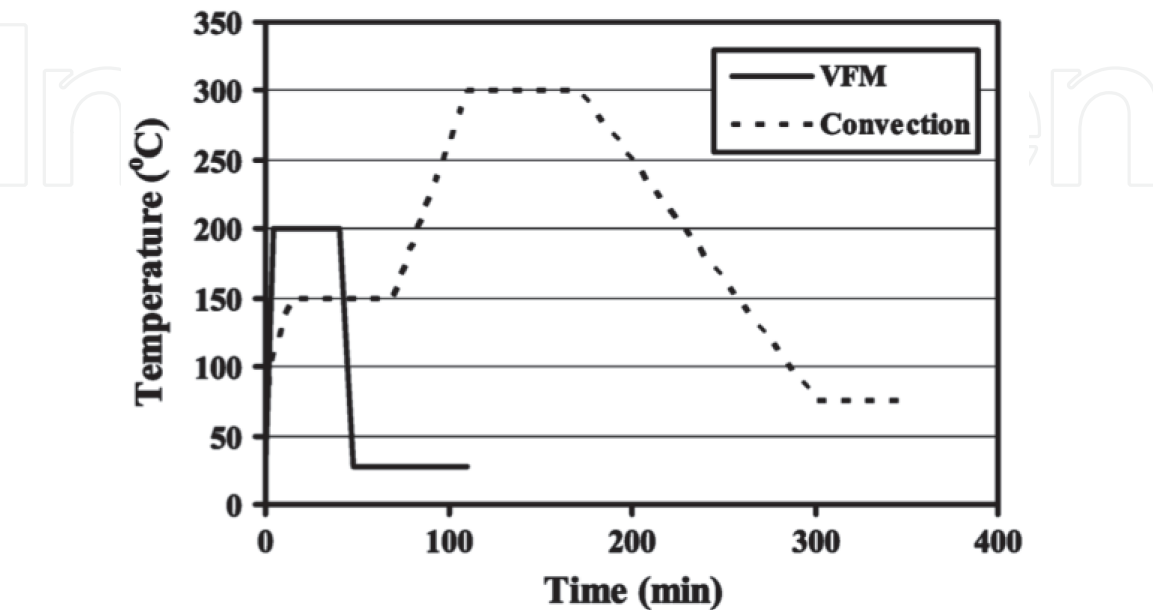


Figure 12.
Typical cure temperature-time profile of polymers using VFM and conventional thermal curing (reproduced from [26]).

Aromatic polyimide	T _g (°C)	Weight loss (%)	σ (S/cm)	ε _r	tanδ	E _{BR} (MV/cm)	CTE (ppm/°C)	E' (GPa)	E'' (MPa)	T _{BR} (MPa)	S _{BR} (%)	Moisture absorption (%)
PMDA/ODA	377–399	3%/100 h at 325°C	< 10 ^{−18}	3.1–3.5	10 ^{−3} –10 ^{−2}	4.5 (1 μm) 3.5 (10 μm)	22–40	3.0		170	40–110	1.3–3.5
BPDA/ODA							43	4.1	140	230	73–110	
BTDA/ODA	279				6 × 10 ^{−3}	1.8 (20 μm)	40	1.0		150	15	1.5
PMDA/PPD							2	12.2	400	296	5	
BPDA/PPD	477–500			2.9–3.1	2 × 10 ^{−3}		3–7.5	8.8–10.2	334–400	390–600	15–47	1.0
BTDA/PPD							30	7.1	232–400	248	18	
ODPA/PPD							35	8.1	251–350	263	18	
BTDA/ODA-MPD	320		10 ^{−20}				40–47	3.3				
PMDA-BPDA/ODA-PPD							19	4.72		260	44	
PMDA-BPDA/PPD (alternate)							2.3	9.6		254	7	
PMDA-BPDA/PPD (random)							5.9	9.1		248	10	
PMDA-BTDA/PPD (alternate)							9.3	8.4		189	4	
PMDA-BTDA/PPD (random)							15.2	7.4		216	9	
PMDA-ODPA/PPD (alternate)							6.8	8.1		223	10	
PMDA-ODPA/PPD (random)							20.8	5.3		184	17	
IPDA/MPD				2.8								
BTDA/DAPF	358			3.1–3.4								

Fluoroninated polyimide	T _g (°C)	Weigth loss (%)	σ (S/cm)	ε _r	tanδ	E _{BR} (MV/cm)	CTE (ppm/°C)	E' (GPa)	E'' (MPa)	T _{BR} (MPa)	S _{BR} (%)	Moisture absorption (%)
6FDA/ODA	290											
6FDA/MPD				3.0								
6FDA/PPD							48	3.8		108	6	
6FDA/DABTF	300			2.58								
6FDA/20FMDA	189	10% at 455°C		2.6								
6FDA/Rf _b MPD	257	5% at 472°C		2.7			86	1.7		72	6	0.5
6FDA-PMDA/TFMOB-PPD	>400			2.6–2.8			29	9.8			22	
3FXDA/ODA				2.8								
3FXDA/Rf _b MPD	394	5% at 465°C		2.5			67	1.9		115	25	1.1
6FXDA/ODA				2.8								
6FXDA/Rf _b MPD	347	5% at 458°C		2.3			70	2.0		116	28	0.6
6FXDA/TFMB	420			2.4			6					12
6FXDA/TFMOB	375			2.8			10					8
PMDA/3FDAM	420–430											

T_g: Glass transition temperature.
σ: Electrical conductivity; ε_r: Dielectric constant; tanδ: Dielectric loss factor; E_{BR}: Dielectric strength. Data given at 25°C.
CTE: coefficient of thermal expansion; E': Young modulus; E'' Mechanical loss modulus; T_{BR}: Tensile at breakdown; S_{BR}: Elongation at mechanical breakdown.

Table 3.
Main physical properties of aromatic and fluorinated polyimides (data taken from [8]).

Microwave heating of polymers occurs because of dielectric loss mechanisms. When an external electric field is applied to a dielectric material, three types of polarization can occur [24]. These ones are the electronic, ionic or atomic, and orientational or dipolar polarization mechanisms. The main coupling mechanism between microwave radiation and polymer dielectrics is through dipole orientation under the applied electric field. The efficiency of coupling microwave energy into a polymer depends on different factors, which include the dipole strength, the dipole mobility and the dipole mass.

The VFM curing principle is to subject the deposited films to very high frequency waves (>1 GHz) causing the macromolecular structure to vibrate (see **Figure 11**). The vibrations then locally heat the coating which thus polymerizes. The advantages of the VFM curing method are the short annealing time. It is around 10 minutes only compared to a few hours for a conventional full thermal cycle. Moreover, the lower associated temperature ($\sim 200^{\circ}\text{C}$) during the application of microwaves enables shorter curing cycles (see **Figure 12**). The industrial interest could be to replace standard thermal curing techniques with faster and lower cost VFM method.

3. Polyimide physical properties ... at a glance

Polyimides are therefore materials which have very good thermal, electrical and mechanical properties. These depend essentially on their chemical structure generated by the choice and the reaction of the basic monomers constituting them.

This chapter ends by a summary of the physical properties of the main aromatic and fluorinated polyimides that have been developed over the last decades, as shown in **Table 3**.

4. Conclusion

This chapter has introduced polyimide materials which are now since few decades commonly used as dielectrics or insulating materials in the electronics and high voltage engineering industries for different purposes. It has been reviewed the state-of-the-art on the polyimide thermal stability. Moreover, the synthesis and imidization reactions, the main precursor monomers, the different deposition and process techniques, and the curing methods were presented. Finally, the main physical properties were summarized. This will offer a good overview as an introduction for the rest of this book.

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