EMAC 276 Polymer Properties and Design

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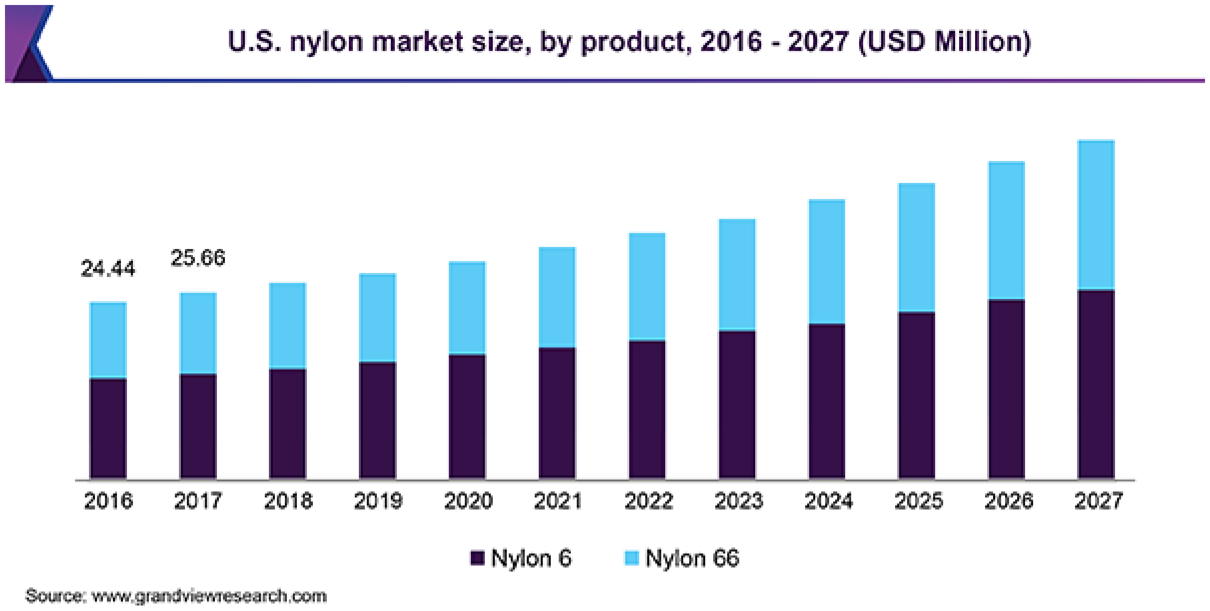
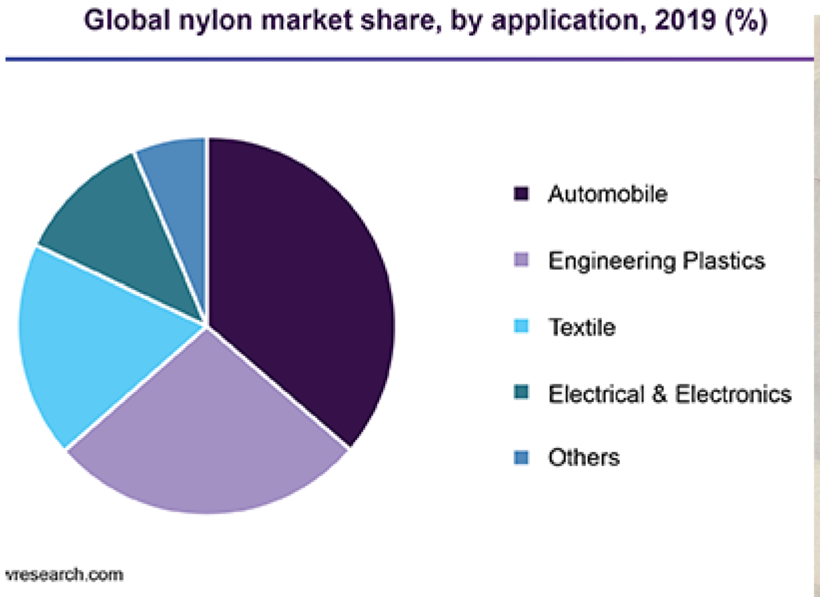
Polyamides or Nylons





https://www.grandviewresearch.com/industry-analysis/nylon-6-6-market

Industrially Important Nylons (Aliphatic Polyamides):

1. nylon 4,6: $\text{+}NH(CH_2)_4NH{-}CO(CH_2)_4CO\text{+}_n$, poly(tetramethylene adipamide);
2. nylon 6,6: $\text{+}NH(CH_2)_6NH{-}CO(CH_2)_4CO\text{+}_n$, poly(hexamethylene adipamide);
3. nylon 6,9: $\text{+}NH(CH_2)_6NH{-}CO(CH_2)_7CO\text{+}_n$, poly(hexamethylene azelamide);
4. nylon 6,10: $\text{+}NH(CH_2)_6NH{-}CO(CH_2)_8CO\text{+}_n$, poly(hexamethylene sebacamide);
5. nylon 6,12: $\text{+}NH(CH_2)_6NH{-}CO(CH_2)_{10}CO\text{+}_n$, poly(hexamethylene dodecanedioamide);
6. nylon 6: $\text{+}NH(CH_2)_5CO\text{+}_n$, polycaprolactam;
7. nylon 11: $\text{+}NH(CH_2)_{10}CO\text{+}_n$, poly(11-aminoundecanoic acid);
8. nylon 12: $\text{+}NH(CH_2)_{11}CO\text{+}_n$, poly(12-aminododecanoic acid).

n-Nylons:
nylon 6, nylon 11, nylon 12

Nylon m,n:
m – diamine and *n* – diacid

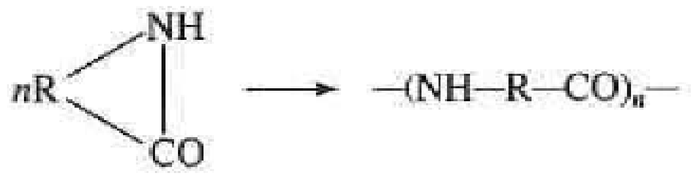
Copolymers:
nylon 6,6/6, nylon 6,6/6,10

Nylon 6,6: 1935, DuPont, Carothers, et al.
Nylon 6: 1939, I.G. Farbenindustrie (Germany), P. Schlack

Typical Nylon Syntheses

n-Nylons (from α,ω-aminoacid):

a) $$n\,H_2N{-}R{-}COOH \longrightarrow {-}(NH{-}R{-}CO)_n{-} + n\,H_2O$$

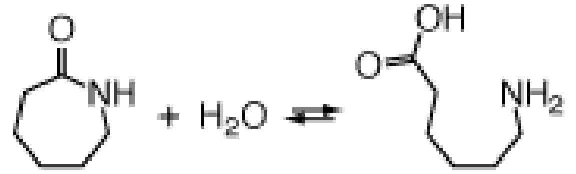
b) 

Nylon m,n: (from nylon salt)

$$n\,H_2N{-}R{-}NH_2 + n\,HOOC{-}R_1{-}COOH \rightarrow$$
$$-(HN{-}R{-}NH{-}OC{-}R_1{-}CO)_n{-} + n\,H_2O$$

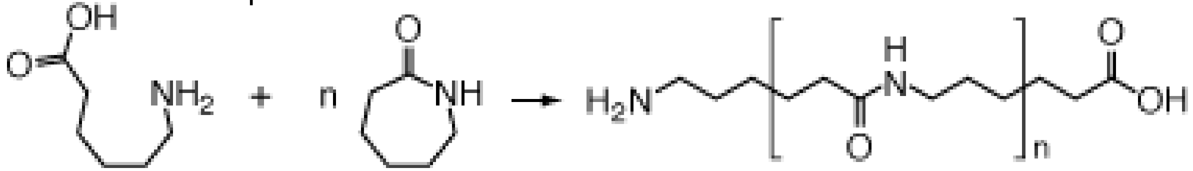
Nylon 6:

Method (a): Hydrolytic ring opening polymerization



caprolactam
m.p. 69.3°C

ω-amino caproic acid



Method (b): Anionic ring opening polymerization

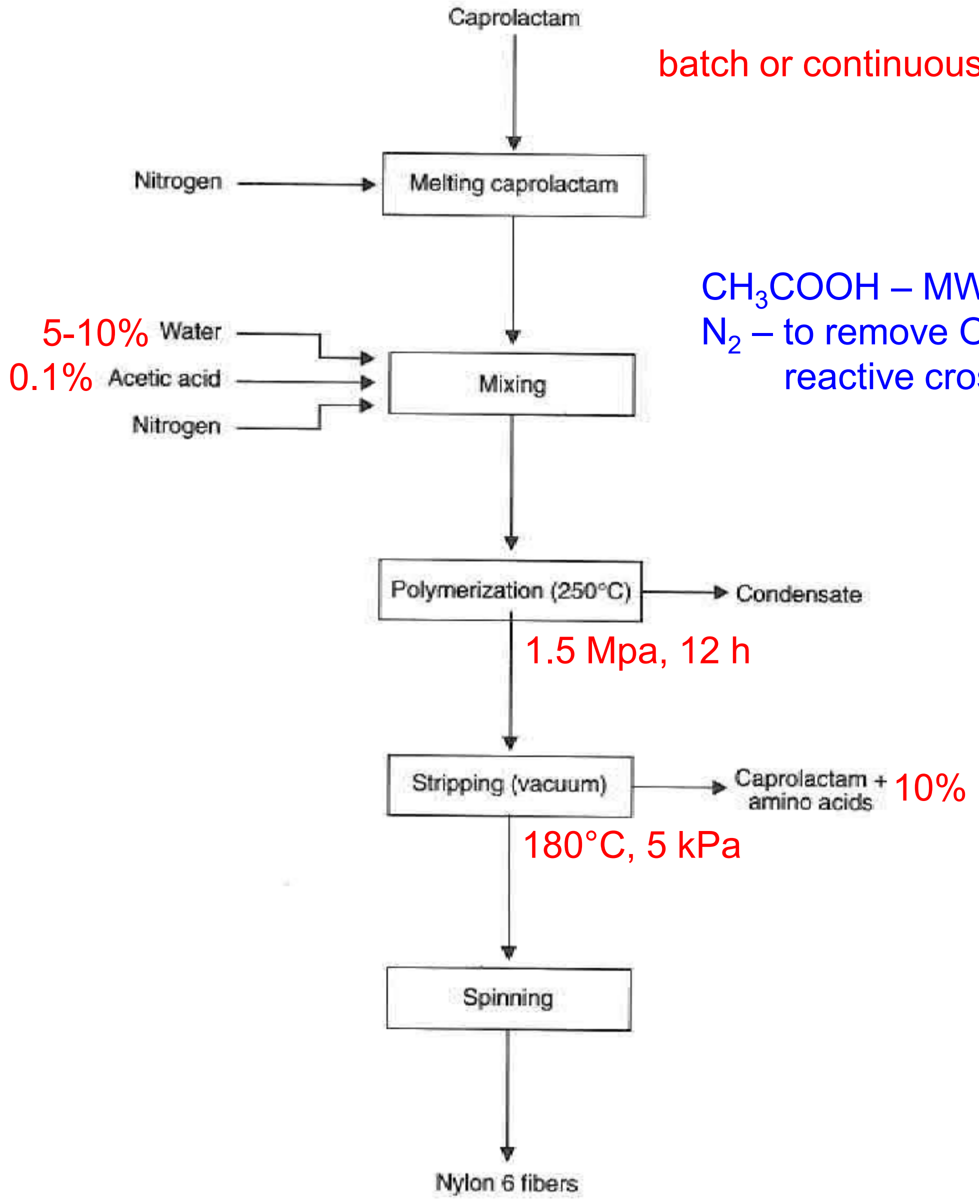


Figure 5.2 Hydrolytical polymerization of caprolactam.

batch or continuous processes

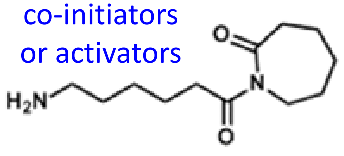
CH_3COOH – MW regulator
N_2 – to remove O_2 and avoid reactive crosslinking

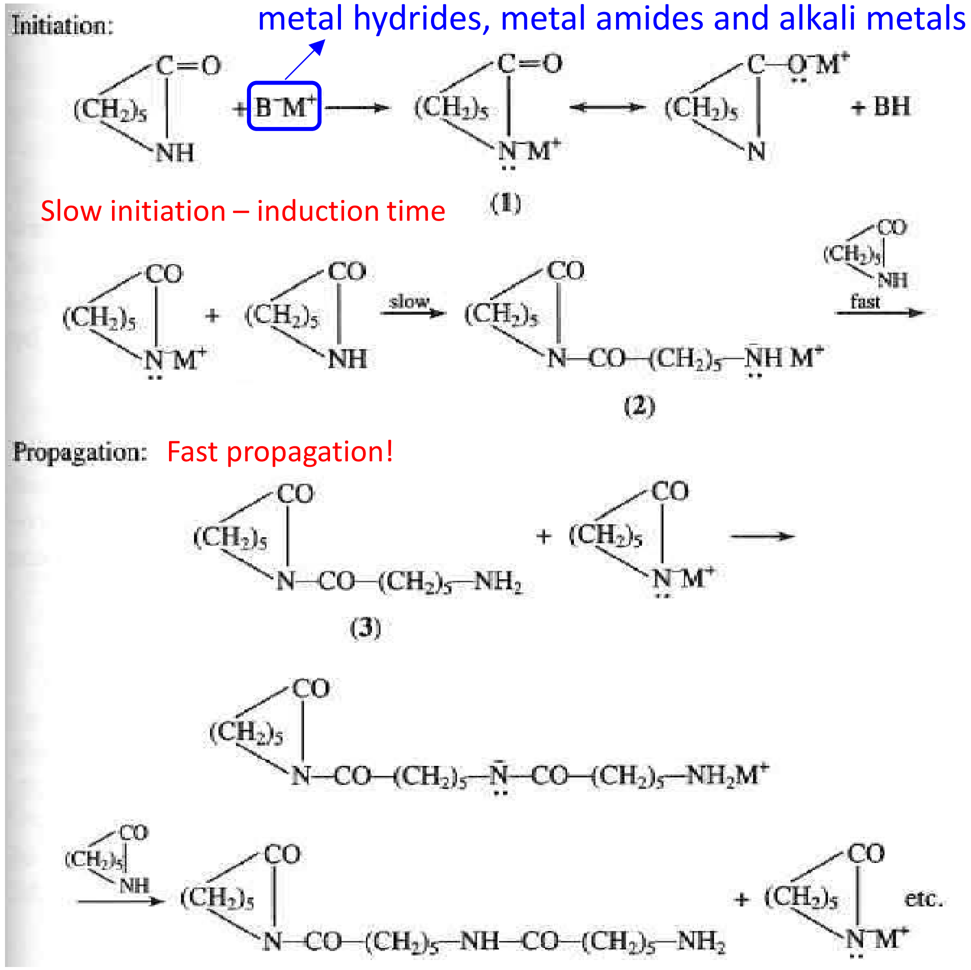
Anionic Ring Opening Polymerization

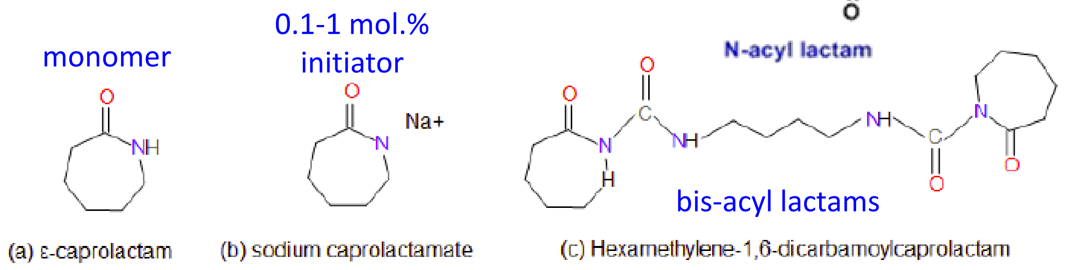
Method (b): Anionic ring-opening polymerization for rapid reactive injection molding (RIM)

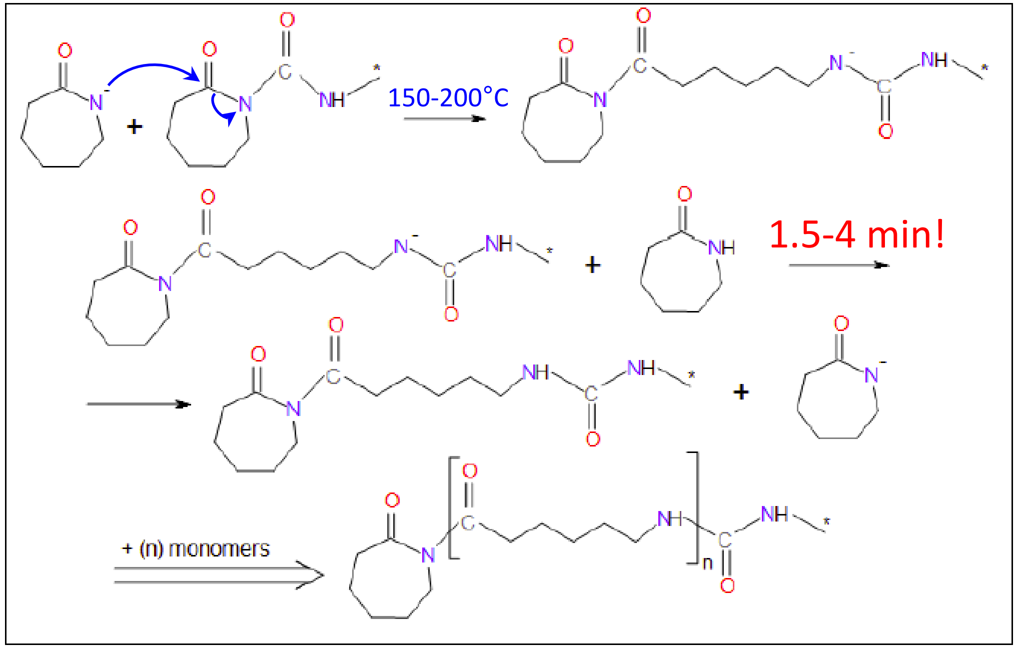
Strategy to eliminate induction time:

0.15-0.5 mol.% co-initiators or activators



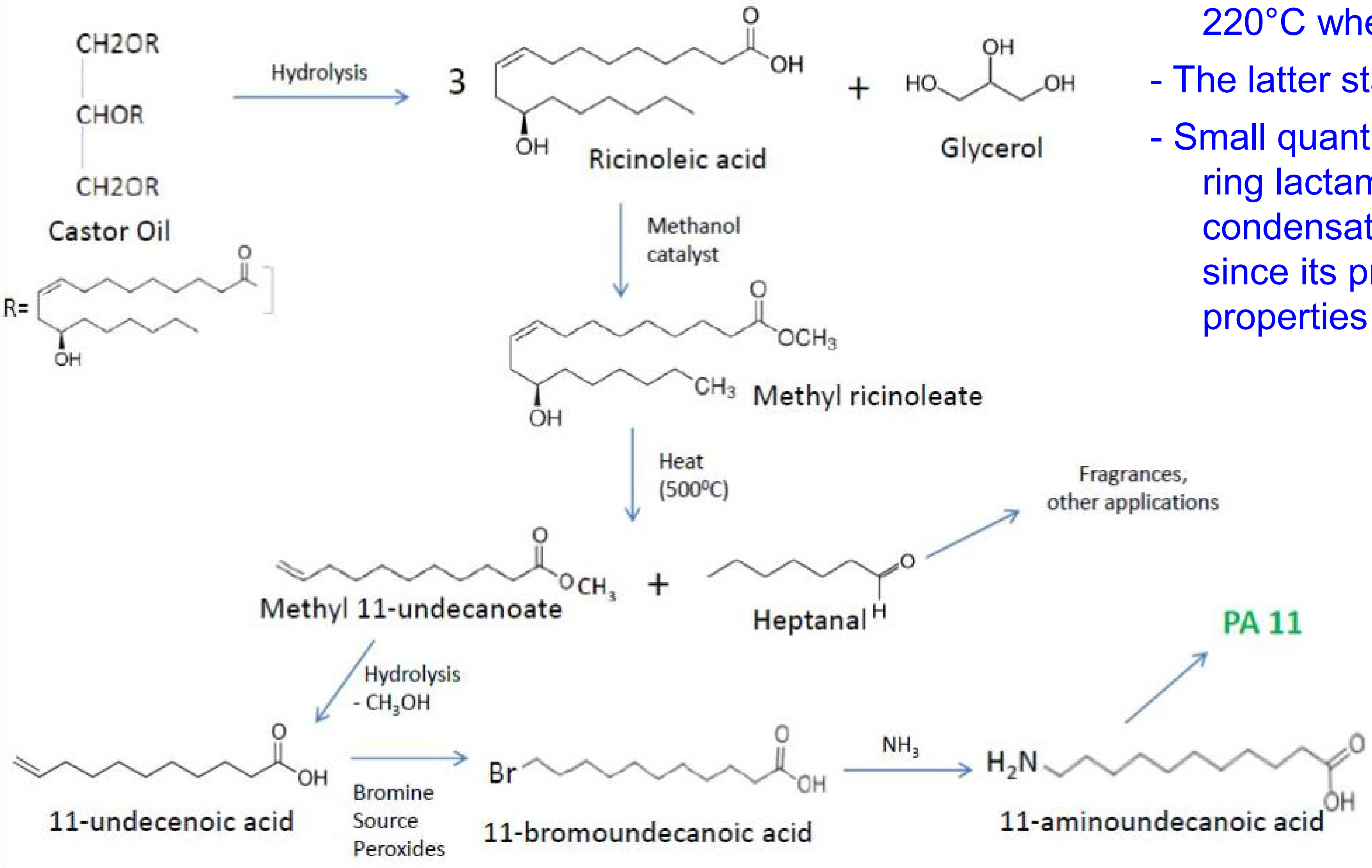






Biosource-based Nylon 11

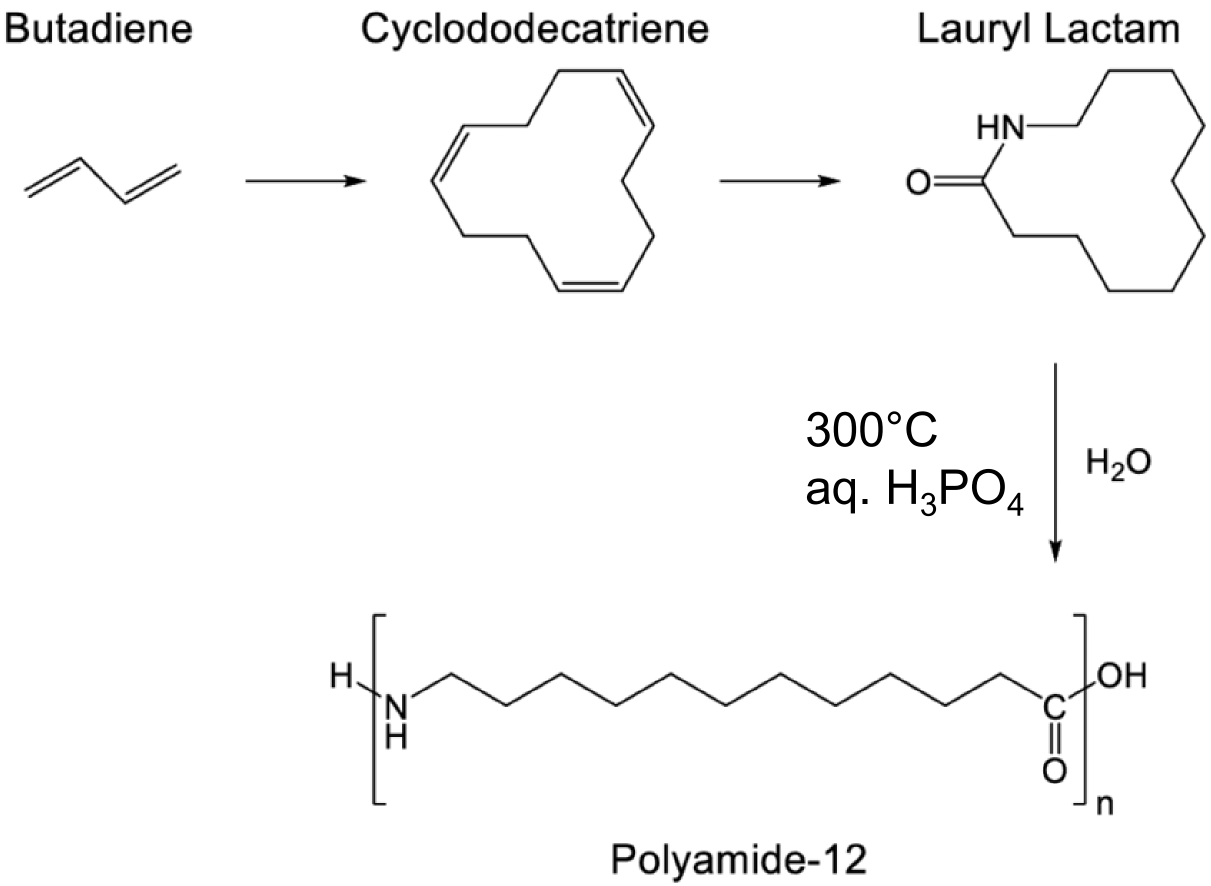
From Castor Oil to Nylon 11 (Arkema)



- Self-condensation in a continuous process at 220°C when withdrawing H_2O continuously.
- The latter stages under reduced pressure.
- Small quantities of 0.4-0.6% of 12-membered ring lactam are produced by intramolecular condensation but it is not normally removed since its presence has little effect on the properties of the polymer.

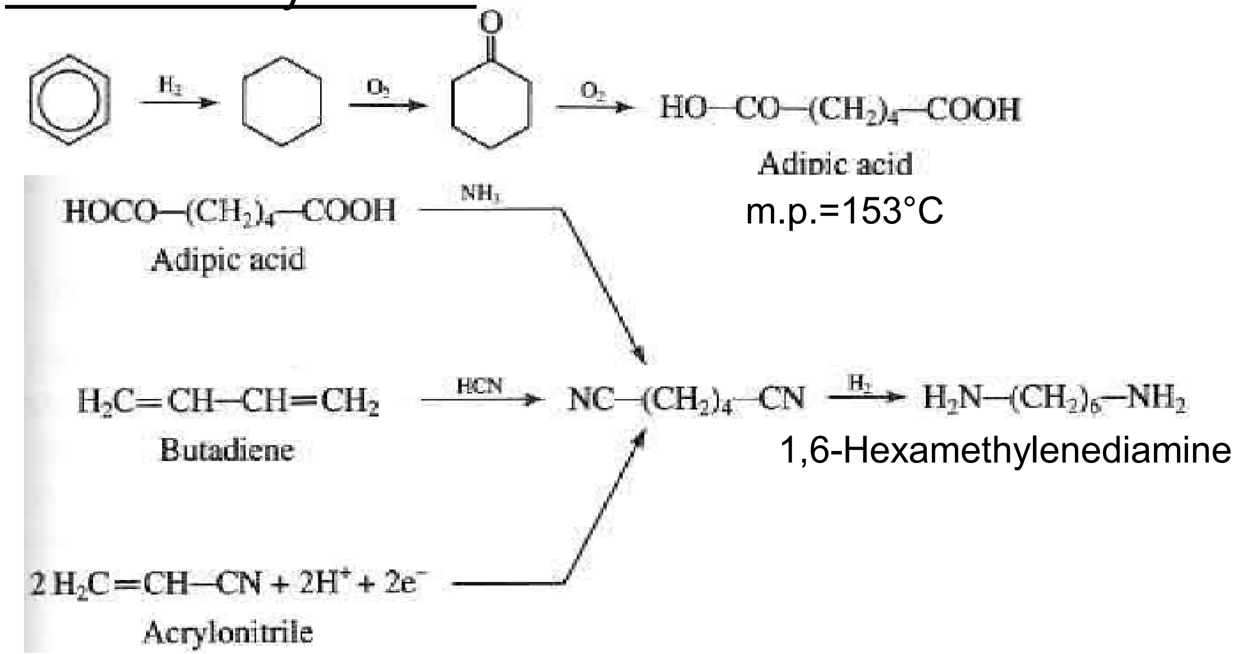
Nylon 12

Hydrolytic ROP of Laurolactam (Evonik)



Nylon 6,6

Monomer Synthesis



Polycondensation via Nylon Salt

$$HOOC-(CH_2)_4-COOH + H_2N-(CH_2)_6-NH_2 \longrightarrow$$

$$[^-OOC-(CH_2)_4-COO^-][^+H_3N-(CH_2)_6-NH_3^+]$$

Nylon salt Crystallized from methanol

$$\longrightarrow -\!\!\left[CO-(CH_2)_4-CO-NH-(CH_2)_6-NH\right]_n + 2H_2O$$

- Acetic acid as MW regulator.
- 220°C at 2 MPa for 1-2 h.
- Then 270-280°C to complete the polymerization.

Properties of Nylons

Strong hydrogen-bonding (HB) in both amorphous and crystalline phases. HB prevents chain sliding in the crystals, avoiding creep.

These structural characteristics determine the general properties of nylons such as:

Advantages

- high melting points - high heat deflection temperatures;
- high tensile strength, rigidity, hardness and resistance to creep;
- high abrasion resistance;
- low linear coefficient of thermal expansion (CTE);
- good solvent/base resistance due to low solubility;
- good oxygen barrier but poor CO_2 barrier – food packaging;
- various nylon copolymers with tailored properties (e.g., reduced T_m and crystallinity).

Disadvantages

- tendency to absorb water from the environment, e.g., 14% for nylon 4,6 and 8-9% for nylon 6,6/nylon 6. nylon 11 and 12 absorb less moisture;
- low melt viscosity and oxidative crosslinking above T_m;
- poor UV resistance (need stabilizers);
- easily attacked by strong acids and oxidizing agents;
- volume shrinkage after crystallization and happens over a long period of time;
- electrical and mechanical properties affected by moisture content.

Properties of Commercial Nylons

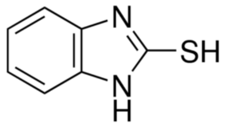
Table 5.1 General properties of nylons

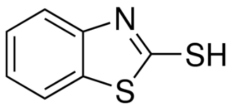
Property	*4,6*	*6,6*	*6,9*	*6,10*	*6,12*	*6*	*11*	*12*	*6,6/6,10 (35:65)*	*6,6/6,10/6 (40:30:30)*
Density (g/cm^3)	1.18	1.14	1.09	1.09	1.07	1.13	1.04	1.02	1.08	1.09
Melting temperature (°C)	280	264	205	215	210	220	185	175	195	160
Heat distortion temperature (°C)										
– at 0.5 MPa	–	199	–	160	160	160–180	150	140	–	–
– at 1.8 MPa	150–170	100–104	60	66–85	65	54–91	55	50	–	30
Tensile strength (MPa)	100	80	59	59	59	76	38	46	38	52
Tensile modulus (GPa)	3	3	–	2.1	–	2.8	1.4	1.4	1.4	1.4
Elongation at break (%)	30	80–100	–	100–150	150–300	50–100	280–300	200	>200	300
Izod impact strength (J/m)	–	27–53	59	85–107	53–101	53	96	101	107	–
Rockwell hardness	R123	R118	–	R111	R114	R112	R108	R107	–	R83
Water absorption at saturation (%)	14	8.0	–	2.4	3	9.0	2.0	1.8	6.5	10.7
Coefficient of linear expansion (10^{-5} cm/cm·°C)	9.2	10	–	15	9	9.5	15	12	–	–

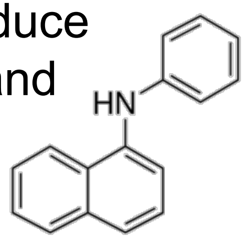
Additives to Nylons

1. Nucleating agents

Fine silica or phosphorus compounds to reduce crystallite size with improved impact strength and abrasion resistance.







2. Heat stabilizers

For good resistance to high temperature aging, mercaptobenzimidazole, mercaptobenzothiazole, phenylnaphthylamine, phosphoric acid esters, and effective copper salts (to prevent oxidative crosslinking).

3. Light stabilizers

Carbon black improves outdoor UV resistance.

4. Impact modifiers

Rubber-type materials such as polyurethanes, ethylene-propylene rubbers, ionomers, ABS polymers, acrylates and methacrylates. Rubber droplets are dispersed in the nylon matrix of nylon.

5. Other fillers

Plasticizers, lubricants, flame retarders, pigments, fungicides, etc. depending on their specific applications. The most frequently used halogen-free flame retarder is based on melamine derivatives.

6. Reinforcing fillers

Glass fibers, glass beads, carbon fibers or certain mineral fillers such as talc. They impart a significant increase in stiffness, impact tensile strength, hardness as well as in heat deflection temperature and maximum service temperature. Self-extinguishing properties of glass-filled nylons are also improved.

Table 5.2 Effect of glass fiber reinforcement on properties of nylon 6 and 6,6

	Nylon 6		*Nylon 6,6*	
Property	*Unfilled*	*Filled*	*Unfilled*	*Filled*
Tensile strength (MPa)	76	115–220	80	160
Elongation (%)	50–100	2–3	80–100	3–5
Flexural modulus (MPa)	2410	5000–11 000	3000	8000
Izod impact strength (J/m)	53	60–190	53	–
Heat deflection temperature under load (°C)				
– 0.5 MPa	160–180	220	200	250
– 1.8 MPa	54–90	190–215	100–104	250
Coefficient of linear expansion (10^{-5} cm/cm · °C)	9.5	–	9.9	2.8
Water absorption at saturation (%)	9	–	8–9	5–6
Maximum service temperature (°C)	180	199	199	241

Applications of Nylons

Different Forms

- Fiber applications
  - 50% into tire cords (nylon 6 and nylon 6,6)
  - rope, thread, cord, belt, and filter cloths
  - Monofilaments - brushes, sports equipment, and bristles (nylon 6,10)
- Plastics applications
  - bearings, gears, cams
  - rollers, slides, door latches, thread guides
  - clothing, light tents, shower curtains, umbrellas
  - electrical wire jackets (nylon 11)
- Adhesive applications
  - hot melt or solution type
  - thermoset reacting with epoxy or phenolic resins
  - flexible adhesives for bread wrappers, dried soup packets, book-binding.

Different Market Categories

- Appliance

  Laundry, cooking, dish washers and disposers, consumer electronics, house cleaning tools, small kitchen appliances, sewing machine; personal care and grooming
- Automotive

  Chassis parts, decorative body parts, functional body parts, electrical parts, fuel system, timing-belt cover, heating, ventilating, air-conditioning accessories (https://www.tytlabs.co.jp/en/review/issue/files/471_045usuki.pdf)
- Business equipment

  Business machines, vending machines, office furniture
- Consumer products

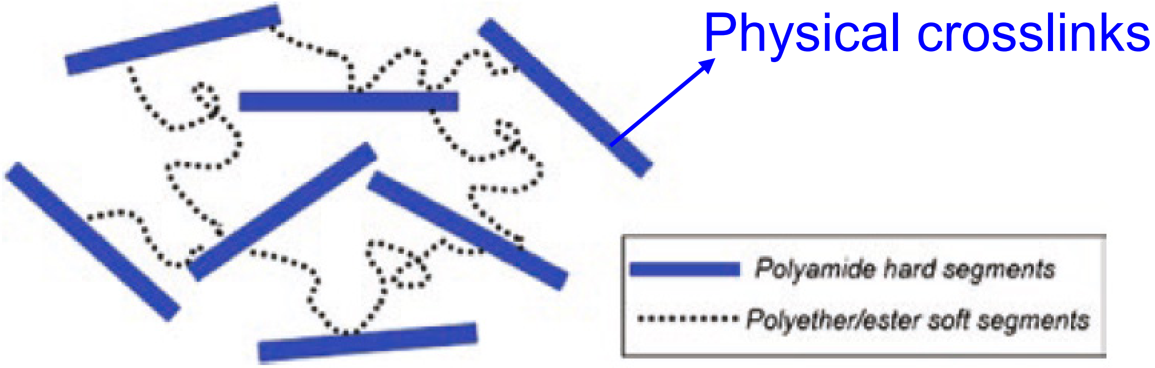
  Kitchen utensils, toys, sporting goods, apparel fitments, personal accessories, photographic equipment, musical instruments, brush bristles, film for cooking, fishing line

Applications of Nylons

Market Categories

- Electrical
  Industrial controls, wiring and associated devices; industrial connectors, batteries, telephone parts, switches
- Hardware
  Furniture fittings, door and window fittings, tools; lawn and garden implements; boat fittings
- Machinery
  Agricultural, mining and oil drilling, food processing, printing, textile processing, engine parts, pumps, valves, meters, filters, air blowers, material handling equipment, standard, components, gears, cams, sprockets, bearings, gaskets, pulleys, brushes
- Packaging
  Nylon films in packaging pharmaceutical products and food boil-in-the-bag treatment (nylon 11), chemical/abrasion resistant coatings.

Polyamide Thermoplastic Elastomers (TPEs)



Poly(ether-*b*-amide) (PEBAX) block copolymers

$$\left[NH-(CH_2)_5-CO \right]_x O \left[(CH_2)_4-O \right]_y$$

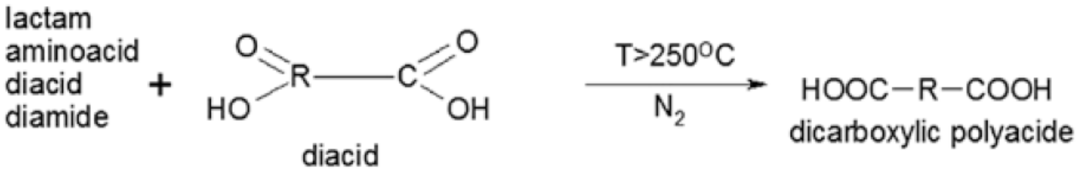
Hard segment   Soft segment

Hard segment (physical crosslinks):
PA12, PA6, PA11
Soft segment (flexibility):
PTMO (or PTHF), PEO, PPO

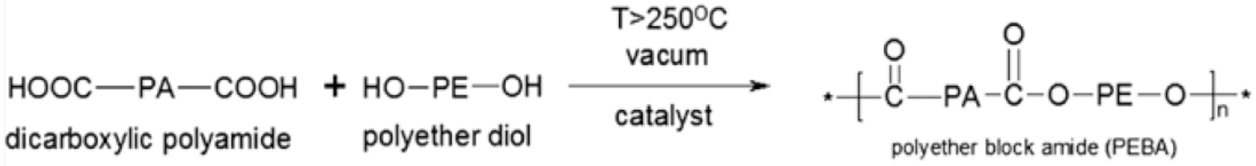
Synthesis

Direct polycondensation in the melt

Polyamide (MW=300-15000) w/ dicarboxyl end groups



Polymeric diol (MW = 100-6000)



Polyamide Thermoplastic Elastomers (cont'd)

The **properties** of polyamide thermoplastic elastomers are controlled by the appropriate selection of the type, the length and the ratio of the hard and soft blocks.
Abrasion resistance is as good as in the case of polyurethanes. UV stability is excellent even without stabilizers.

Applications include automobile bumpers and fascia, housings for business machines, gaskets and seals, watch straps, high performance athletic goods, military shoes, etc.

Table 5.3 Some properties of polyamide thermoplastic elastomers

Property	*Value*
Density (g/cm^3)	1.01–1.15
Melting temperatures (°C)	120–270
Maximum service temperature (°C)	150
Tensile strength (MPa)	18–42
Tensile modulus at 300% (MPa)	18–33
Elongation (%)	270–500
Hardness (Shore)	60A–63D
Flexural modulus (MPa)	20–410
Water absorption (% vol.)	1.2–120

PEBAX Elastomers

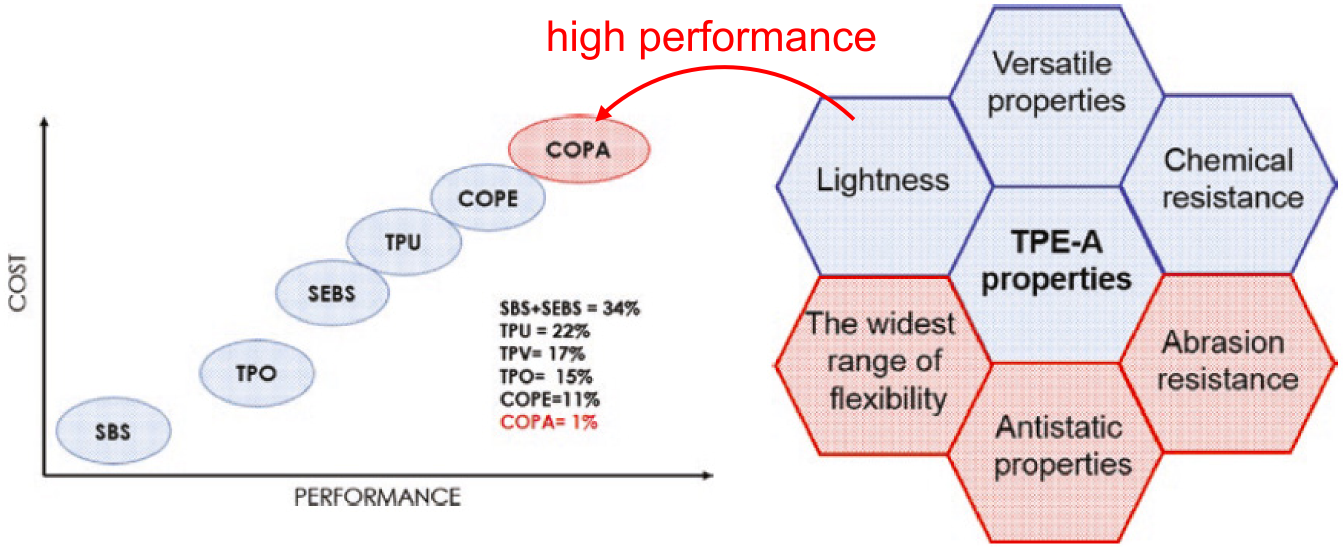


Table 1 Commercial TPE-A

Supplier	Country	Trade name	Hard segments	Soft segments
ARKEMA	France	Pebax® Pebax®Rnew	PA12/PA6 PA11	PTMO/PPO PTMO
Evonik	Germany	Vestamid®E	PA12 PA612	PTMO
EMS-Grivory	Switzerland	Griflex Griltech®ELY	PA12 PA12	Polyether PTMO
Ube Industries	UK	Ubesta XPA	PA12	Uniquely designed polyether
Brüggemann Chemical	Germany	Nyrim®	PA6	–

Table 2 Applications of Vestamid® grades produced by Evonik

Vestamid®	Typical applications
E40-S3	Noiseless gears, seals, functional elements of sports shoes, processing aids in the extrusion of TPU, films
E47-S1 E47-S3	Sports shoe soles, packaging films, non-skid surfaces, sports glasses, protective goggles
E55-S1 E55-S3	Alpine ski boots, sports shoe soles, pneumatic lines, rolls, films
E58-S4	Specialty grade for transparent sports shoe soles
E62-S1	Alpine ski boots, noiseless gears, conveyor belts
E62-S3	Alpine ski boots, noiseless gears, conveyor belts, fiber optic cable
EX9200	Decorative and protective films

Table 3 Applications of Pebax® grades produced by ARKEMA

Grade	Main properties	Typical applications
Pebax®	High performances independent of temperature	Power transmission belts and silent gears, transportation, sport, and leisure
Pebax®Clear	High transparency High UV resistance	Racket bumpers, sport, and ski shoes
Hydrophilic Pebax®	Permanent antistatic properties, high moisture and vapor transmission rate	Permanent antistatic additive, breathable film in construction wrapping, medical, sport clothing, food packaging
Pebax®MED	Biocompatibility sterilization feasibility	Angioplasty and urology catheters, connectors for catheters, medical gown
Pebax®Rnew	Bio-based origin and high performance	Air freshener, conveyor belts, fibers, power transmission belts, racket bumpers, sport shoes

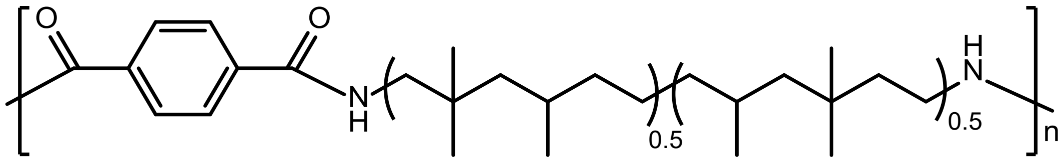
Latko P., Boczkowska A. (2016) Electronic Applications of Polyamide Elastomers and Its Composites. In: Ponnamma D., Sadasivuni K., Wan C., Thomas S., Al-Ali AlMa'adeed M. (eds) Flexible and Stretchable Electronic Composites. Springer Series on Polymer and Composite Materials. Springer, Cham

Partially Aromatic Polyamides

At least one of the components is aromatic:

1) Aromatic (phthalic) diacid rather than aromatic diamine;
2) If aromatic diamine, diisocyanate route is used;
3) Amorphous to semicrystalline polyamides are obtained.

I. Poly(trimethylhexamethylene terephthalamide) [nylon 6(3)T]

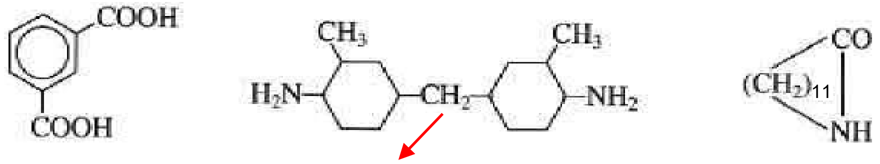


Amorphous polymer due to copolymerization, highly transparent, high T_g – heat resistance, rigid and hard.

Table 5.4 Some properties of poly(trimethyl hexamethylene) terephthalamide

Property	*Value*
Density (g/cm^3)	1.12
Tensile strength (MPa)	68
Tensile modulus (MPa)	3000
Elongation (%)	70
T_g (°C)	150
Deflection temperature at 1.8 MPa (°C)	130
Molding shrinkage (cm/cm)	0.007
Refractive index	1.566

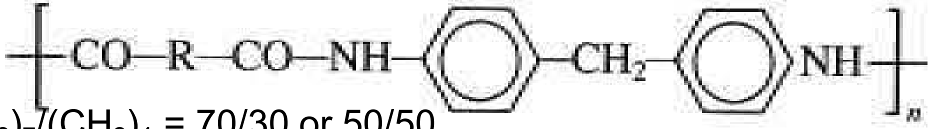
II. Copolymers based on:



bis-(4-amino-3-methylcyclohexyl)methane laurolactam

Very low water absorption and the lowest density (1.06 g/cm^3).

III. Copolymers from 4,4'-diphenylmethane diisocyanate (MDI):



R = $(CH_2)_7/(CH_2)_4$ = 70/30 or 50/50

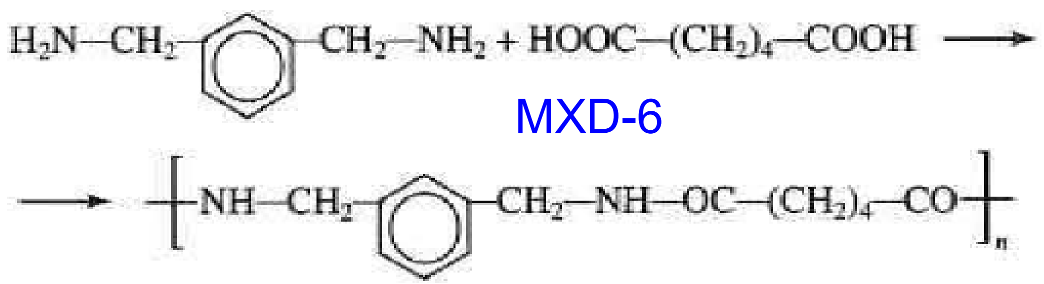
Strong, similar to PC or PSF. Used for transparent flow meter parts, sight glasses and spectacle frames, counters and containers for solvents, transparent housings for different equipment, X-ray apparatus windows and sanitary fittings

Table 5.5 Some properties of MDI based polyamides

	Value	
Property	*Polyamide 70/30*	*Polyamide 50/50*
Density (g/cm^3)	1.17	1.16
T_g (°C)	140	180
Heat deflection temperature at 1.8 MPa (°C)	127	160
Tensile strength (MPa)	83	82
Tensile modulus (MPa)	1869	2289
Elongation at break (%)	80	30
Mold shrinkage (cm/cm)	0.005	0.0035

Partially Aromatic Polyamides (cont'd)

IV. MXD-n (n = 6-12) (based on *m*-xylylene diamine)



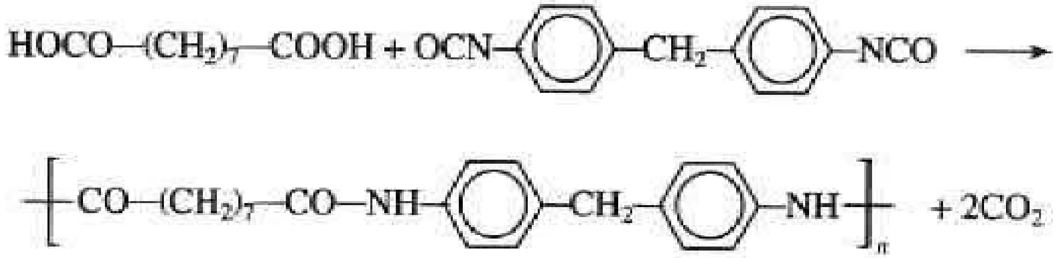
MXD-6

T_m ~235-240°C, T_g = 100°C, low heat deflection temp. about 96°C. Glass fiber composites with enhanced mechanical property and heat resistance. Used for shafts and gears, electrical pings and sockets, machine components and other mechanical parts.

Table 5.6 Some properties of glass filled composites

Property	*Value*
Tensile strength (MPa)	185
Flexural modulus (GPa)	10
Heat deflection temperature at 1.8 MPa (°C)	220–230
Molding shrinkage (cm/cm)	0.003–0.006

V. Copolymers based on MDI and azelaic acid



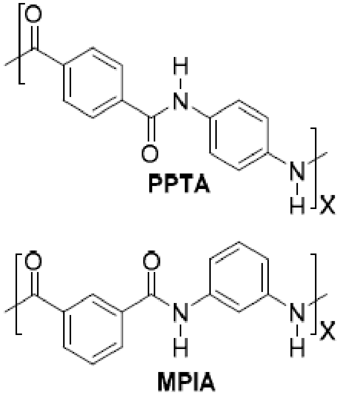
T_m at 290°C and T_g at 135°C. High modulus and tensile strength, high heat resistance, dimensional stability, good electrical properties and excellent chemical resistance. It is used in automotive under-the-hood and other harsh industrial applications.

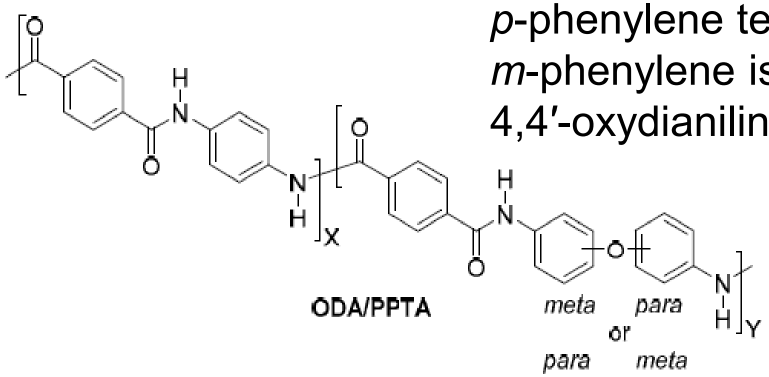
Table 5.7 Some properties of unfilled and glass reinforced semicrystalline polyamides

		Glass reinforced	
Property	*Unfilled*	*30% glass*	*40% glass*
Density (g/cm^3)	1.17	1.40	1.49
Tensile strength (MPa)	71	148	195
Tensile modulus (MPa)	1681	5757	7193
Elongation (%)	5.0	4.0	4.1
Flexural modulus (MPa)	2459	7648	10018
Heat deflection temperature at 1.8 MPa (°C)	130	247	247

Aromatic Polyamides or Aramids

Structure, Aramid Type, Brand Names and Company of Commercial Aramids





p-phenylene terephthalamides (PPTA)
m-phenylene isophthalamides (MPIA)
4,4′-oxydianiline (ODA)

Aramid type			Brand names	Company
PPTA	MPIA	ODA/PPTA		
X			Kevlar®	DuPont, Wilmington, DE, United States
	X		Nomex®	
X			Twaron®	Teijin, Arnhem, the Netherlands
	X		Teijinconex®	
		X	Technora®	
X			Heracron®	Kolon Industries, Seoul, Korea
X			Alkex®	Hyosung, Seoul, Korea
	X		*Meta* Aramid Yarn & Thread®	SRO Group, Heatherbrae, New South Wales, Australia
X			Taparan® *Para*-aramid	Yantai Tayho Advanced Materials Co., Ltd, Yantai, Shandong, China
	X		Newstar® *Meta*-Aramid	
	X		Arawin®	Toray Chemical Korea Inc., Seoul, Korea
	X		Metaone	Huvis, Seoul, Korea

Polymers **2017**, *9*, 414/1-44; doi:10.3390/polym9090414

Aromatic Polyamides (cont'd)

Solution polycondensation or Interfacial Polycondensation

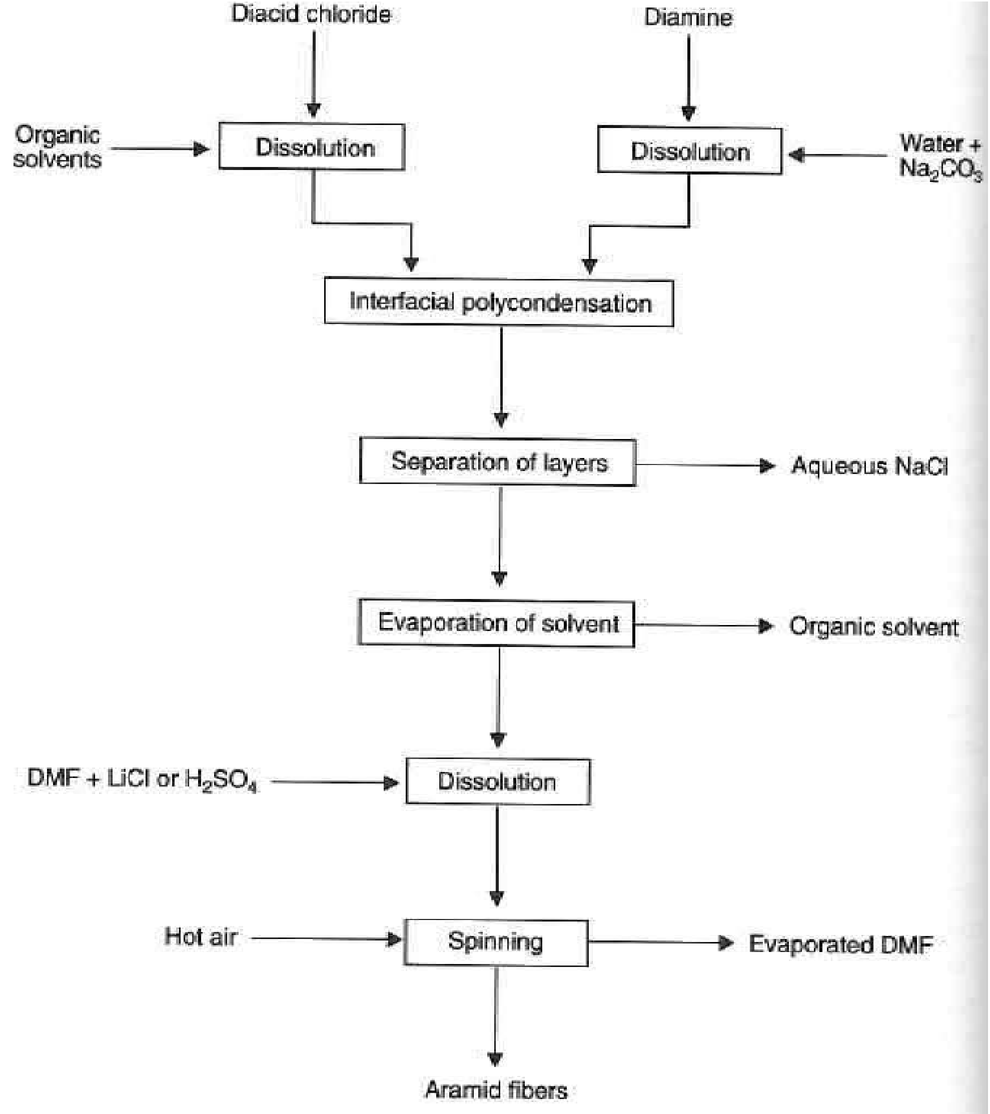
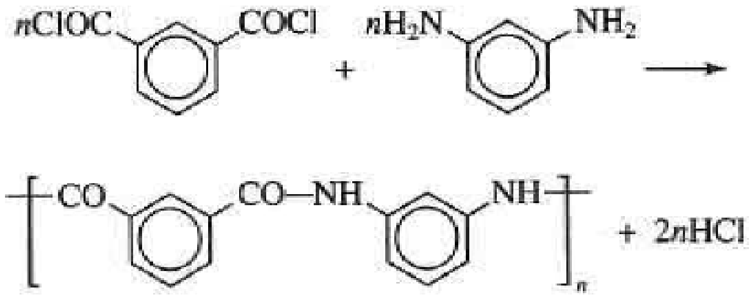


Figure 5.5 Preparation of aramid fibers by interfacial polycondensation.

I. Poly(*m*-phenylene isophthalamide) (1967)



Dr. Wilfred Sweeny at DuPont invented NOMEX

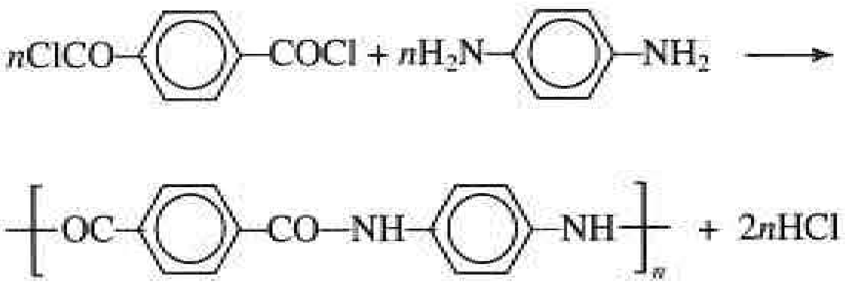
Synthesis
Monomers dissolved in N,N'-dimethyl acetamide (DMAc) at -10 °C under rapid stirring for 1 h.

Properties
Fibers have good mechanical properties at elevated temperatures. At ambient temperature, their mechanical, chemical and electrical insulation properties are similar to those of nylon 6 and 6,6. At 260°C, they still retain half of these properties. T_m is 368-390°C; the material resists ignition but shrinks considerably.

Applications
Heat protective clothing and electrical insulation at high temperatures.

Aromatic Polyamides (cont'd)

II. Poly(*p*-phenylene terephthalamide)



Solution polycondensation: 2:1 mixture of hexamethylphosphoramide and N-methylpyrrolidone at -10°C

Properties

- The highest strength/modulus properties among commercially available fibers (only drop to ½ at 250 °C).
- Better electrical insulation than glass fibers.
- Won't melt, only form char at 425 °C.

Table 5.8 Comparative tensile properties of aramid fibers and glass fibers

Property	*Aramid fiber*[a]	*Glass fiber*
Density (g/cm^3)	1.44–1.47	2.49–2.55
Tensile strength (GPa)	3.45–3.62	3.10–4.14
Tensile modulus (GPa)	124–186	72–85
Elongation at break (%)	2.0–2.5	4.3–4.8

[a] Aramid = poly(*p*-phenylene terephthalamide)

History

- In 1964, Kwolek persuaded her technician to spin fibers from the lyotropic solution (in sulfuric acid) and found strong fibers.
- In 1971, modern Kevlar was introduced; however, she was not very much involved in the development of various applications of Kevlar.

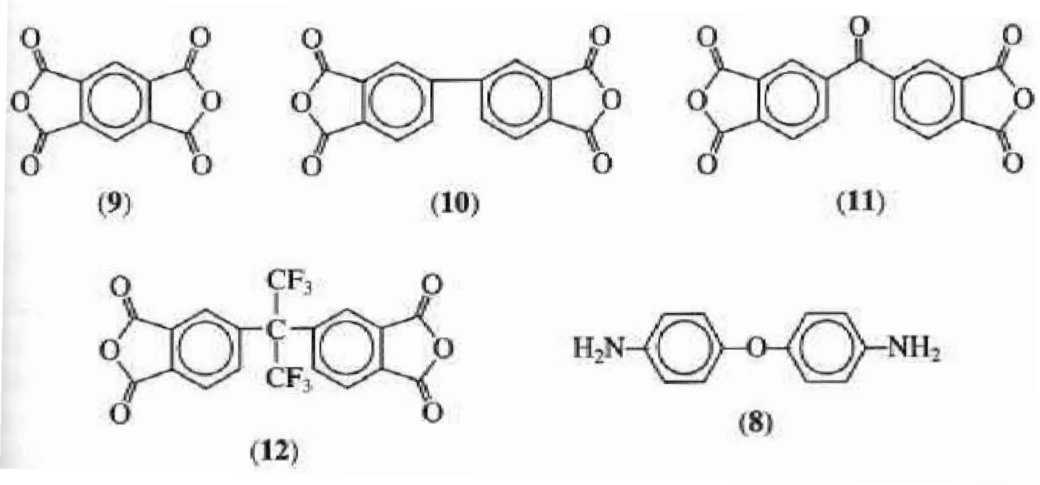
Inventor of Kevlar, Stephanie Kwolek, an American chemist of Polish origin

Applications

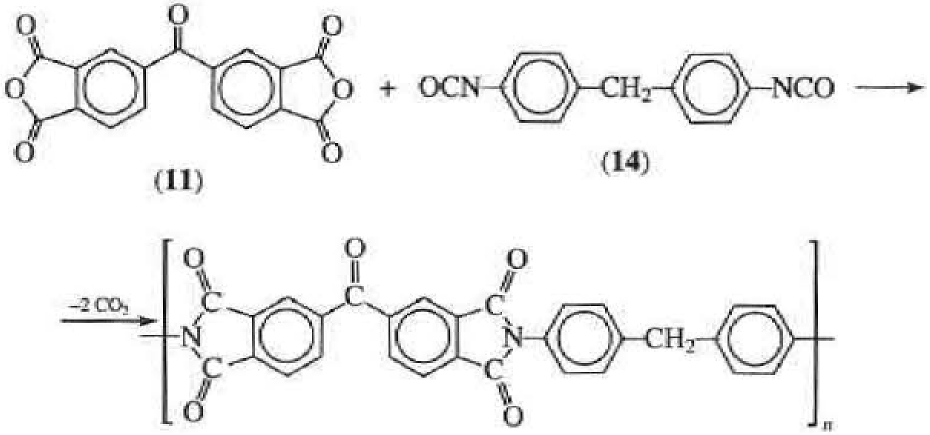
- Fibers and textiles (the ultimate bullet-proof vests).
- Used as fillers for epoxy composites, competing with glass, carbon, and steel fibers, to replace aluminum.
- Boat hulls, military protection helmets and filament-wound rocket.

Polyimides

Unmodified Polyimides

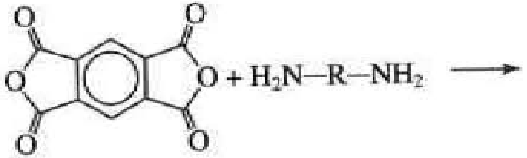


One-step synthesis from diisocyanate

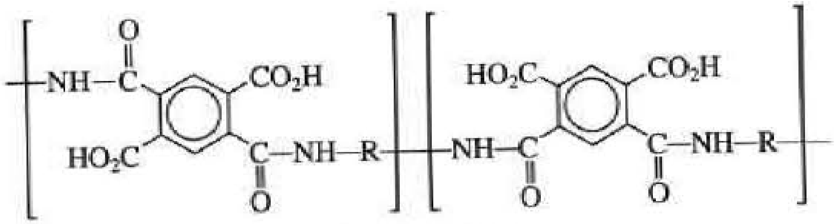


CO_2 can make polyimide foam for acoustic insulation of jet engines

Two-step synthesis

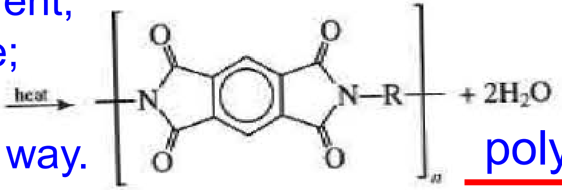


DuPont Kapton®
R = **(8)**,
T_g~380°C, no
T_m detected!



Polyamic acid (13)

Curing in solid-state:
- 150-200°C to remove solvent;
- stepwise to 300 °C to cure;
- Important to completely remove H_2O in a controlled way.



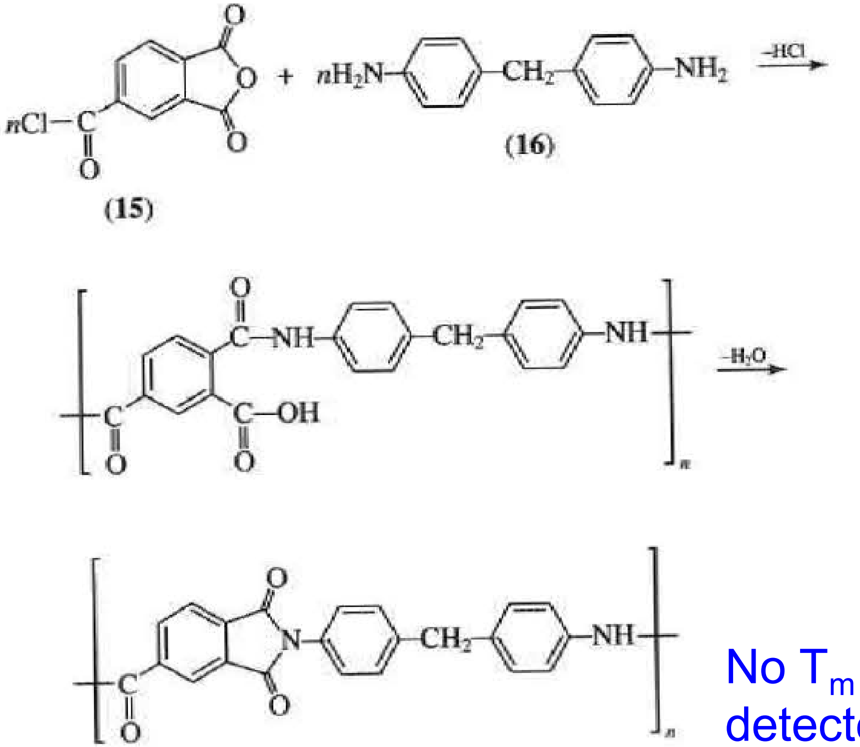
polyimide

Properties
- Heat resistance as high as 357°C. After 1000 h exposure to air at 300°C, 90% tensile and dielectric strength retain.
- Excellent abrasion resistance, flame, solvent and oxidative degradation resistance as well as high energy radiation resistance.

Applications
- Insulating enamels and varnishes for electric motors and cables and for coating glass cloth.
- Compounded with graphite, molybdenum disulfide, PTFE and metal powders and piston rings for jet engines.
- Flexible electronics.

Modified Polyimides

1. Poly(amide-imides) (PAIs)



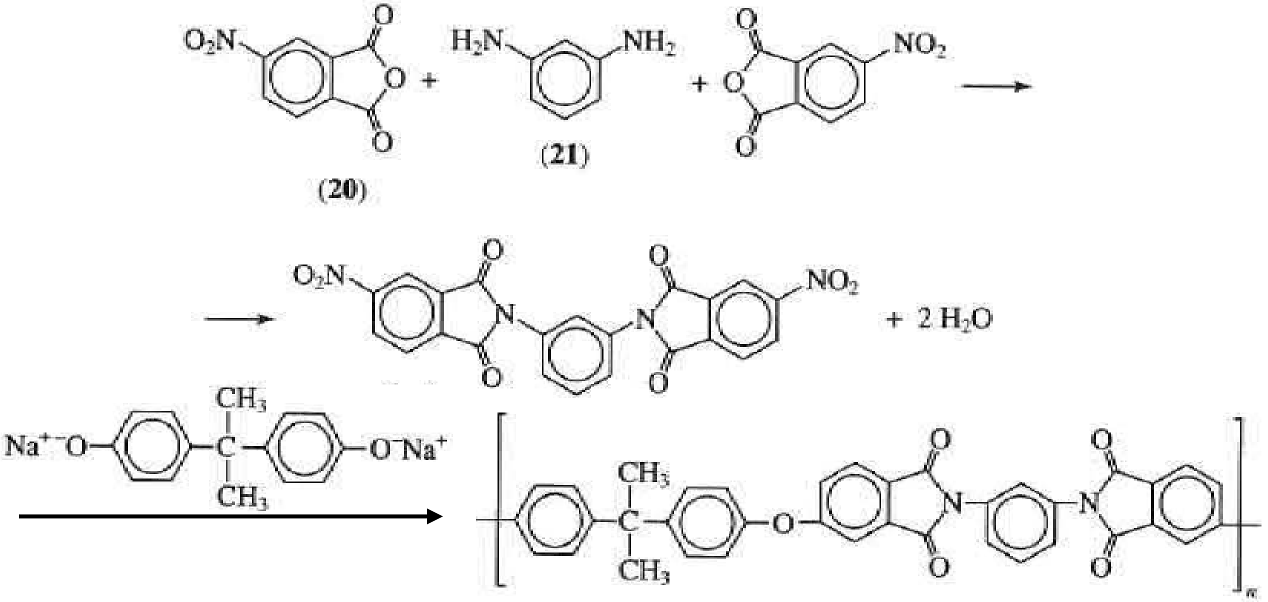
No T_m detected!

Table 5.9 Some properties of poly(amide-imides)

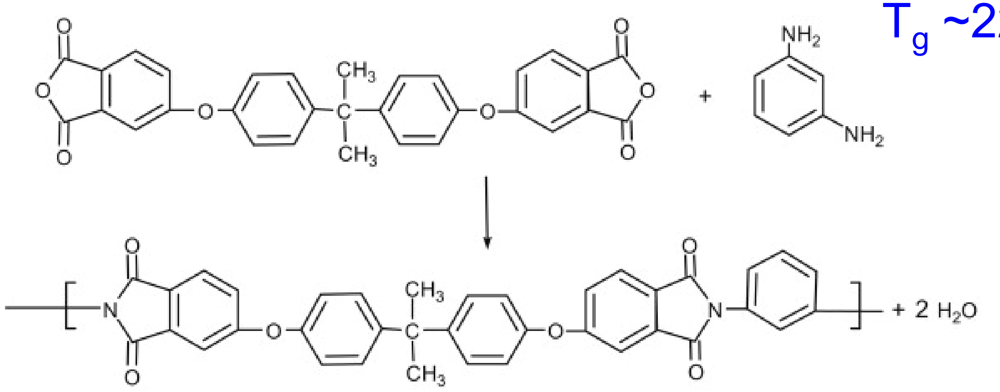
Property	*At ambient temp.*	*at 260°C*
Density (g/cm^3)	1.14	–
Glass transition temperature, T_g (°C)	275	–
Heat deflection temperature at 1.82 MPa (°C)	270	–
Continuous use temperature (°C)	200	–
Tensile strength (MPa)	185	50
Flexural modulus (GPa)	4.55	3.00
Elongation (%)	12	22

2. Poly(ether-imides) (PEIs) – Ultem® by GE (SABIC)

Nitro-displacement polymerization



Imidization polymerization



Amorphous
T_g ~220°C

PEIs

Properties

- Amorphous thermoplastic resin with amber transparency
- High deflection temperature (200°C at 264 psi), high tensile strength and flexural modulus (480,000 psi), and very good retention of mechanical properties at elevated temperatures
- Good electrical properties, which remain stable over a wide range of temperature and frequencies
- Good UV-light resistance and weatherability
- Inherently flame resistance without the use of additives
- High limiting oxygen index of 47 and the lowest specific optical density of any unfilled thermoplastic
- Resistant to alcohols, acids, and hydrocarbon solvent but dissolves in partially halogenated solvents
- Good hydrolytic stability
- Most of the PEI grades has a UL94 flame resistance rating of VTM-0, is FDA compliant, EU Food Contact Compliant, and ISO10993 compliant in natural color

https://omnexus.specialchem.com/selection-guide/polyetherimide-pei-high-heat-plastic

Applications

- Automotive and Aerospace Market

  Transmission components, throttle bodies, ignition components, thermostat housings, bezels, reflectors, lamp sockets, and electromechanical systems such as fuses, gears, bearings, solenoid bodies, ignition switches and oil pump drives

- Electrical and Electronic Market

  Electrical switches and controls, electrical motor parts, printed circuit boards, and connectors

- Disposable & Re-Usable Medical Applications
- Metal Replacing Property for Industrial Applications & Appliances

  HVAC equipment and fluid handling systems, manufacture of institutional kitchenware, microwave cookware, steam and curling irons, dual-ovenable trays for food packaging