

Curing Agents for Epoxy Resin

Introduction

Epoxy resin was discovered in 1938 by Pierre Castan, a chemist in Switzerland. As of 1989, 137,000 tons of epoxy resin had been produced in Japan, and epoxy resin has been used in a wide range of fields, such as paints, electricity, civil engineering, and bonds. This is because epoxy resin has excellent bonding property, and also after curing, it has excellent properties on mechanical strength, chemical resistance, electrical insulation. In addition, epoxy resin is able to have various different properties as it is combined and cured together

with various curing agents.

This issue describes the types of curing agents for epoxy resin and their characteristics comparing to Three Bond products.

The epoxy resin compositions of Three Bond currently on the market are the Three Bond 2000 Series (base agent for epoxy resin), the Three Bond 2100 Series (curing agent for epoxy resin), and the Three Bond 2200 Series (one-part thermal cure epoxy compound resins).

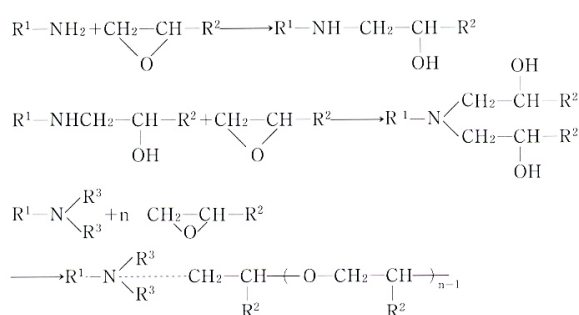
Contents

Introduction.....	1
1. Amines.....	2
1-1. Aliphatic amine.....	2
1-2. Aromatic amine.....	4
1-3. Modified amines.....	4
2. Polyamide resin.....	5
3. Tertiary and secondary amines.....	5
4. Imidazoles.....	6
5. Polymercaptan curing agent.....	6
5-1. Liquid polymercaptan.....	6
5-2. Polysulfide resin.....	6
6. Anhydrides.....	8
7. Latent curing agents.....	8
7-1. Boron trifluoride-amine complexes.....	8
7-2. Dicyandiamide.....	8
7-3. Organic acid hydrazide.....	9
8. Photo- and ultraviolet-curing agents.....	9
Conclusion.....	10

1. Amines

Amine compounds are classified into primary, secondary, and tertiary amines, in which one, two, and three hydrogen molecule(s) of ammonia (NH₃) have been substituted for hydrocarbon, respectively. Amines are called monoamine, diamine, tri-amine, or polyamine according to the number of amines in one molecule. Amines are classified into aliphatic, alicyclic (Three Bond 2106), and aromatic amines according to the types of hydrocarbons involved, and the all are important curing agents for epoxy resin.

Aliphatic amine (Three Bond 2103) is curing



In general, curing agent must have more than three active hydrogen atoms and two amino groups in a molecule so that the cured resin becomes crosslinked polymer, according to the reaction of the above equation. The loading of the curing agent in epoxy resin becomes optimal when the number of moles in epoxy groups is equal to that of active hydrogen.

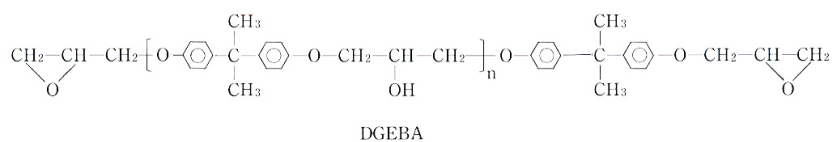
The curing speed of individual amines depends on the type and loading of amine, and the type of epoxy resin. The most commonly used glycidyl-ether type resins easily cure at room

temperature. The cured resin has excellent properties, and its heat resistance is 100°C. Aromatic amine has been developed to achieve greater heat resistance and chemical resistance than those of aliphatic amine.

The curing of epoxy resin by amine curing agents is expressed by the formula shown below; the active hydrogen in primary amine reacts with an epoxy group to form secondary amine, and the secondary amine reacts with an epoxy group to cure. Then, the resultant tertiary amine polymerizes epoxy groups.

temperature, but inner epoxy type such as cyclohexene oxide and epoxidized polybutadiene is hardly cured. Glycidyl-ester type cures quite faster than glycidyl-ether type. Diglycidyl ether of bisphenol A (DGEBA), which is a condensation product of bisphenol A and epichlorohydrin, is primarily cured by aliphatic amines at room temperature, but is slowly cured by aromatic amines and requires thermal curing.

Table 1 shows the properties and performances of representative polyamines used as curing agents for epoxy resin.



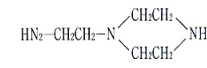
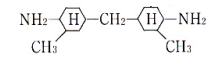
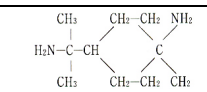
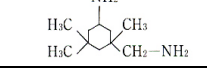
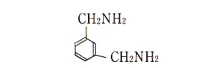
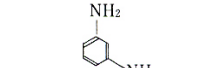
1-1. Aliphatic amine (Three Bond 2103)

Aliphatic amine, which rapidly reacts with epoxy resin, is a representative room-temperature curing agent. However, it generates a large quantity of heat and has a short pot life (usable time). Loading of amines containing no tertiary amine is made at the exact or very closed amount that is said in stoichiometry, and use amount of amines containing tertiary amine is made less than that. If latter curing is performed at high temperature, properties of curing agents that cure at

room temperature are improved. The heat-deformation temperature (HDT) of cured object of DGEBA is 120°C at the highest.

Resins that have been cured using aliphatic amines are strong, and are excellent in bonding properties. They have resistance to alkalis and some inorganic acids, and have good resistance to water and solvents, but they are not so good to many organic solvents. Aliphatic amine irritates the skin and possesses toxicity. Although those that have high molecular weight and low vapor pressure are less toxic, good cautions for handling are required.

Table 1. Properties and performance of amine based curing agents

Class	Subclass	Name of curing agent	Appearance	Viscosity cps (25°C)	Active hydrogen equivalent (amine value)	Loading amount to liquid epoxy resin (phr)	Specific gravity (g/ml) (20°C)	Pot life (100 g) (batch)	Curing conditions		Heat deformation temperature (°C)	Applicability				Remark	
									Temperature (°C)	Time (minutes)		Bonding	Lamination	Casting	Paint		
Aliphatic polyamine	Chain aliphatic polyamine	Diethylenetriamine (DTA)	Transparent liquid	5.6	20.7	5 - 10 Standard value: 8	0.954	20 minutes	Normal to 100	30 minutes to 4 days	115	○	○	○	○	$H_2N-(CH_2)_2-NH-(CH_2)_2-NH_2$	
		Triethylenetetramine (TTA)	Transparent liquid	19.4	24.4	6 - 12 Standard value: 9	0.98	20 to 30 minutes	Normal to 100	30 minutes to 4 days	115	○	○	○	○	$H_2N-(CH_2)_2-NH-(CH_2)_2-NH-(CH_2)_2-NH_2$	
		Tetraethylenepentamine (TEPA)	Transparent liquid	51.9	27.1	7 - 14 Standard value: 12	1.00	30 to 40 minutes	Normal to 100	30 minutes to 7 days	115	○	○	○	○	$H_2N-CH_2-(NHCH_2)_3-NH_2$	
		Dipropenyldiamine (DPDA)	Transparent liquid		29.0	12 - 15		20 to 30 minutes	Normal to 200	30 minutes to 7 days	110	○	○	○	○		
		Diethylaminopropylamine (DEAPA)	Transparent liquid		65.0	4 - 8 Bonding = 8, Casting = 4, Lamination = 6		1 to 4 hours	65 - 115	1 - 4 hours	85	○	○	○	○	$C_2H_5-N(CH_2CH_2CH_2NH_2)_2$	
		AMINE 248	Transparent liquid	1000-3000	42.9	35	0.83	30 minutes	Normal to 100	30 minutes to 4 days	92	○		○		Rare Hexamethylenediamine	
	Alicyclic polyamine	N-aminoethylpiperazine (N-AEP)	Transparent liquid		43	20 - 22	0.984	20 to 30 minutes	Normal to 200	30 minutes to 3 days	103		○	○			
		Lamiron C-260	Transparent liquid	60	31 - 33	31 - 33	0.945		80+150	2 hours + 2 hours	150		○	○	○		
		Araldit HY-964	Transparent liquid	70		15 - 20	0.94	120 minutes	Normal	7 days					○		
		Menthane diamine (MDA)	Transparent liquid	19.0	42.5	22		6 hours	80 - 130 130 - 200	30 minutes to 2 hours 2+3 hours	158	○	○	○			
		Isophoronediamine (IPDA)	Transparent liquid	18.2	41	24	0.924	1 hour	80+150	4+1 hours	149		○	○			
		S Cure 211	Transparent liquid			17 - 21	0.96	30 minutes	Normal to 70	2 hours to 7 days	47	○	○	○	○		
		S cure 212	Transparent liquid			12 - 16	0.98	40 minutes	Normal to 70	2 hours to 7 days	48	○	○	○	○		
		Wandamin HM		m.p 40°C	53	30	0.95		60+150	3+2 hours	150		○	○	○		$NH_2-(CH_2)_6-NH_2$
		1.3 BAC			35.5		0.94										$NH_2-CH_2-(CH_2)_4-CH_2-NH_2$
		Aliphatic aromatic amine	m-xylenediamine (m-XDA)	Crystalline liquid		34.1	16 - 18	1.05	20 minutes	Normal to 60	1 hour to 7 days	115	○				
	Sho-amine X		Liquid	68°	33 - 34	16 - 18	1.05	20 minutes	Normal to 60	1 hour to 7 days	113	○				Xylylenediamine	
	Amine black		Viscous liquid	(50°C) 2000 - 6000		30 - 60	1.20	40 minutes	Normal to 60	1 hour to 7 days		○			○	Xylylenediamine trimer	
	Sho-amine black		Viscous liquid	6000 - 10000		25 - 35	1.18	40 minutes	Normal to 60	1 hour to 7 days	116	○			○	Xylylenediamine trimer	
	Sho-amine N		Liquid	5°	(690)	25	1.18	80 minutes	Normal to 60	1 hour to 7 days	81	○		○		Xylylenediamine derivative	
	Sho-amine 1001		Liquid	100.0		27	1.07		Normal to 60	1 hour to 7 days	73	○		○		Xylylenediamine derivative	
Sho-amine 1010	Liquid		40.0		27			Normal to 60	1 hour to 7 days	70	○		○		Xylylenediamine derivative		
Aromatic amine	Metaphenylene diamine (MPDA)	Solid	mp62 °C	34	14 - 16	0.95	6 hours	80+150	2+4 hours	150	○	○	○				
	Diaminodiphenylmethane (DDM)	Solid	mp8 °C	49.6	25 - 30	1.05	8 hours	80+150	2+4 hours	150	○	○	○		$H_2N-C_6H_4-CH_2-C_6H_4-NH_2$		
	Diaminodiphenylsulfone (DDS)	Solid	mp175 °C	62.1	30 - 35	1.33	Approximately 1 year	110+200 °C	2+4 hours	180 - 190	○	○	○		$H_2N-C_6H_4-SO_2-C_6H_4-NH_2$		

1-2. Aromatic amine (Three Bond 2163)

Aromatic amine has weaker basicity than aliphatic amine and slowly cures at room temperature due to steric hindrance by the aromatic ring. The curing virtually stops in the B-stage of a linear polymer solid due to the large difference in the reaction of primary and secondary amines. Normally, the curing of aromatic amine requires heating in two steps. The first heating is carried out at a rather low temperature of approximately 80°C so as to lessen heat generation, and the second heating is carried out at a high temperature of 150°C to 170°C.

Aromatic amine provides excellent heat resistance, HDT of 150°C to 160°C, and is good in mechanical properties and strong. In addition, the amine has good electrical properties and excellent chemical resistance, particularly against alkalis, and thus it is a curing agent that is highly resistant to solvents.

1-3. Modified amines

The modification of amine curing agents improves the workability as follows:

1. Extends the pot life
2. Increases or decreases the curing speed
3. Improves compatibility with resins
4. Liquefies curing agents
5. Reduces reactivity to carbon dioxide in the air
6. Reduces toxicity and irritation to the skin
7. Decreases weighing error because loading amount is increased

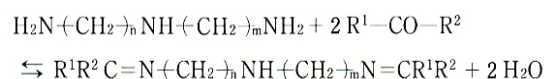
(1) Amine adduct (polyamine epoxy-resin adduct) (Three Bond 2102, 2131B)

When epoxy resin is allowed to react with an excessive amount of polyamine such as DETA and

consumes all of the epoxy groups, an amine adduct having active hydrogen of the residual amino groups is formed. As the adduct has a high molecular weight, it is less volatile, releases less amine odor, is less toxic, and is less exothermic. The loading of the adduct in resins is so much, then the weighing error becomes small.

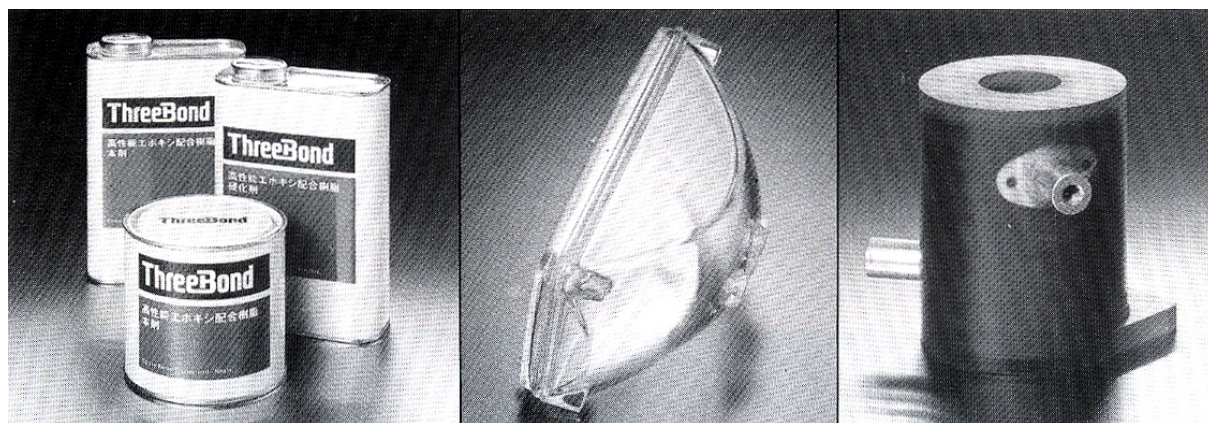
(2) Ketimine (Ketoimine)

Ketimine, which is attracting attention as a curing agent for high solid paints, is formed by the reaction between aliphatic polyamine such as DTA, TTA, DPDA, and m-XDA, and ketone such as methylethyl ketone (MEK) and isobutylketone (MIBK).



Ketimine cures very slowly when mixed with epoxy resin, but it works as a kind of latent curing agent, when it is made to coat or the like, by absorbing moisture in the air and regenerating amines to cure at room temperature.

Practically, ketimine cures at normal temperature in approximately 8 hours, and is used in high solid paints. The cured resin has properties almost same as those of resin cured by the original polyamine, but its application is limited to thin films due to the fact that it regenerates ketones, and its curing speed is slow.



Two-part epoxy-compound resin Adhesion between glass and metal Encapsulation of a coil

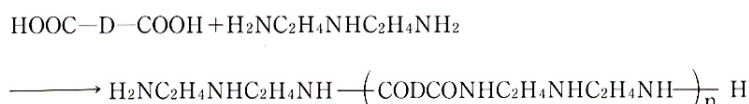
2. Polyamide resin (Three Bond 2105, 2105C, 2105F and 2107) —●

Polyamide resin, which has been widely used as a curing agent for epoxy resin, is formed by the condensation reaction between dimer acid and polyamine, and contains reactive primary and secondary amines in its molecules.

Polyamide amine reacts with bisphenol-A-type epoxy resin to cure at or below normal temperature with moderate heat generation. It cures so slowly

that it has a long pot life.

As polyamide has high hydrocarbon moiety in its molecules, it cures epoxy resin into a highly plasticized rigid thermosetting polymer. The cured resin features high tensile, compression, and bending strengths, while it is stiff, strong, and excellent in shock resistance.

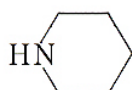


3. Tertiary and secondary amines —●

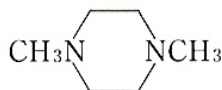
Tertiary amine, the active hydrogen in which has been completely replaced with carbon hydroxide, does not cause an additional reaction with epoxy resin, but works as a polymerization catalyst. Thus, the loading is not constant and depends on the type of curing agent. The curing temperature significantly influences the curing speed, heat generation, and the properties of the cured resin. Thus, the amine is rarely used alone, since,

particularly in large castings, properties at the center and the outer region are different due to the large quantity of heat generation. It is used in the fields of paints and adhesives.

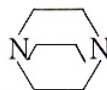
Although tertiary amine is less useful as a curing agent, it is a very important compound as an accelerator for acid anhydrides, and is useful as an accelerator or co-curing agent for polyamine and polyamide curing agents.



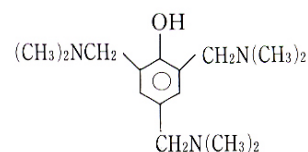
Piperidine



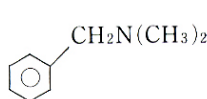
N,N-dimethylpiperidine



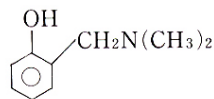
Triethylenediamine



2,4,6-tris
(dimethylaminomethyl)
phenol
(DMP-30)



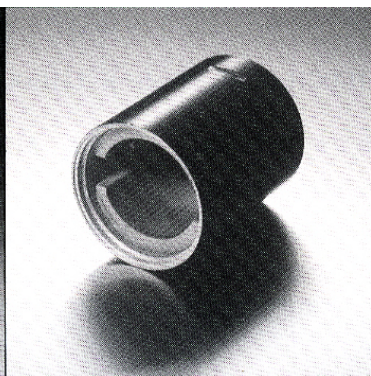
Benzyl dimethylamine
(BDMA)



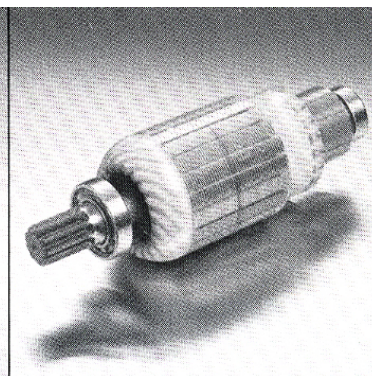
2-(dimethylaminomethyl)phenol
(DMP-10)



One-part epoxy-compound resin



Bonding between ferrite and the yoke



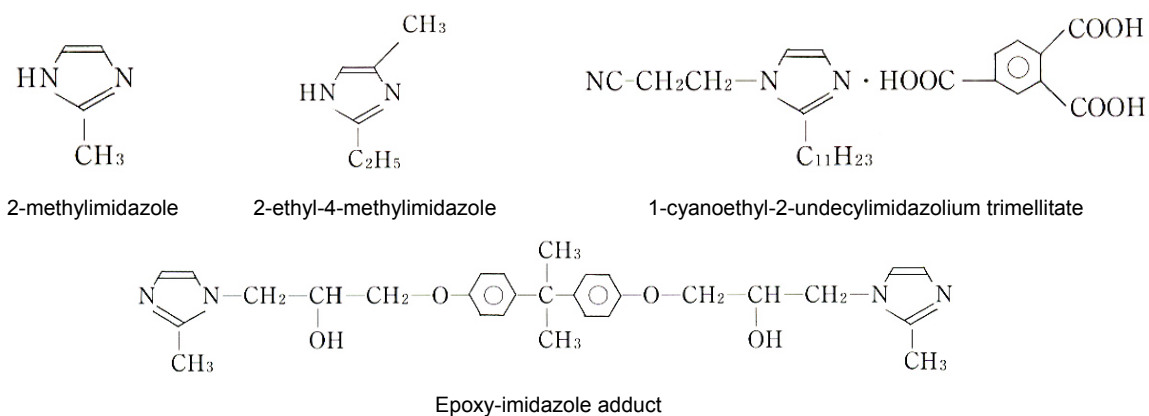
Impregnated locking of the motor coil

4. Imidazoles (Three Bond 2162F, 2163C)

Like tertiary amines such as BDMA and DMP-30, imidazoles are a type of anionic polymerizing curing agent for epoxy resin. Imidazoles are characterized by a relatively long pot life, the ability to form cured resin with a high heat deformation temperature by thermally treating at a medium temperature (80°C to 120°C) for a short time, and the availability of various derivatives having moderate reactivity that improves workability. For example, imidazole carboxylate, epoxy-imidazole adduct, metal salt-imidazole complex compounds, and imidazole that has been reacted with acidic substances are used as curing agents. All are intended to improve workability by achieving a

high pot life and rapid curing at a desired temperature (100°C to 180°C), and have been used in compound resin compositions such as one-part thermosetting coating adhesives, casting materials, and filling materials.

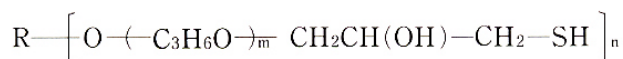
In addition, as in the case of other types of tertiary amine, imidazoles can be used as a curing accelerator or co-curing agent for organic-acid anhydrides, dicyandiamide, polyhydric phenol, and aromatic amine. In such cases, imidazoles exhibit better properties than other types of tertiary amines, including a longer pot life, faster curing speed, and higher heat resistance of the cured substance.



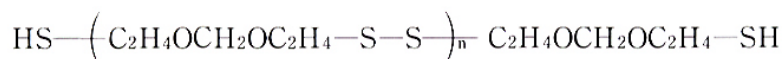
5. Polymercaptan curing agent

5-1. Liquid polymercaptan (Three Bond 2086B Curing Agent)

Polymercaptan, which cures at 0°C to -20°C, is attracting attention as a low-temperature curing agent. It requires to add tertiary amine as an accelerator. At normal temperature, polymercaptan has a pot life of 2 to 10 minutes, and rapidly cures and reaches practical strength in 10 to 30 minutes. The following structure shows a type of polymercaptan curing agent.



5-2. Polysulfide resin (Three Bond 2104)



Like liquid polymercaptan, polysulfide resin has mercaptan groups at its terminals, but the resin does not have low-temperature and fast-curing properties and is used as a curing agent doubling as a flexibilizer. Normally, the resin works as a room-temperature curing agent when used in combination with a tertiary amine or polyamine curing agent. The loading is 50 to 100 wt% and as the loading of polysulfide increases, the cured resin increases in flexibility, shock resistance, and permittivity, while it decreases in curing shrinkage. Due to its good water resistance, polysulfide resin has been used in adhesives, sealing agents, and casting materials.

Table 2. List of acid anhydrides

Type of curing agent	Product name	Appearance	Molecular weight	Acid anhydride equivalent (neutralization equivalent)	Viscosity cps. 25°C	Melting point °C	Specific gravity 25°C	Boiling point °C /mmHg
Phthalic anhydride		Solid	148.1	148	-	130.8	1.527	295/760
Trimellitic anhydride		Solid	192	-	-	168	-	240-245/14
Pyromellitic anhydride		Solid	218	109	-	286	1.68	305-310/30
Benzophenone tricarboxylic anhydride		Solid	322		-	221-255		-
Ethylene glycol bistrimellitate	Rikaresin TMEG	Solid	ca. 410	204	-	64-72	1.46	-
Glycerol tris(trimellitate)	Rikaresin TMTA	Solid	ca. 600	210	-	ca. 70	1.47	-
Maleic anhydride		Solid	98	98	-	52.8	1.48	202/760
Tetrahydrophthalic anhydride	Rikacid TH	Solid	152.1	152	4.65 (105°C)	100<	1.20 (105°C)	120/3
Methyltetrahydrophthalic anhydride	HN-2200	Liquid	-	(81-85)	50-80	-15>	1.21±0.05	-
	Epiclone B-570	Liquid	166	166	ca. 40	-15>	1.201	-
Endomethylene tetrahydrophthalic anhydride	Kayahard CD	Solid	164	164	-	164-165	-	-
Methylendomethylene tetrahydrophthalic anhydride	Kayahard MCD	Liquid	178	-	200-300	10>	1.23 (20°C)	250>
	MHAC-P	Liquid	-	(88-93)	150-300	-	1.23±0.01	-
	MHAC-L	Liquid	-	(87-92)	150-300	-	1.24±0.01	-
Methylbutenyl tetrahydrophthalic anhydride	YH-306	Liquid	234	117	ca. 130	-15>	1.09±0.01	150/1
Dodecyl succinic anhydride	DSA	Liquid	266	-	300-800 (20 °C)	-	1.002 (20°C)	180-182/5
Hexahydrophthalic anhydride	Rikacid HH	Solid	154	154	23.0 (40°C)	34.0<	1.18 (40°C)	110/5
Hexahydro-4-Methylphthalic anhydride	Rikacid MH-700	Liquid	168	161-166	50-70	-15>	1.17	127/5
	Epiclone B-650	Liquid	168	170	ca. 65	-15>	1.17	173/30
	HN-5500	Liquid	168	-	50-80	-15>	1.16±0.01	-
Succinic anhydride	Rikacid SA	Solid	100	100	-	118.0<	1.503	128.2/10
Methylcyclohexene dicarboxylic anhydride	Epiclone EXB-4400	Solid	264	132	-	167	-	-
Alkylstyrene-maleic anhydride copolymer	Smicure MS-1	Solid	-	-	-	ca. 135	-	-
Chlorendic anhydride	Kayahard CLA	Solid	371	371	-	235-239	-	-
Polyazelaic poly(anhydride)	PAPA	Solid	-	-	200 (99°C)	52-65	-	-
	Rikacid PAZ-90	Solid	-	172	1000 (80°C)	55-65	1.19	-

6. Anhydrides (Three Bond 2162G, 2280C)

Anhydrides used as epoxy-resin curing agents have been used as curing agents for electrical insulating materials. Anhydrides require severer curing conditions than amine-based curing agents, but are suitable for making large moldings, as they have a long pot life and form cured resins having relatively well-balanced electrical, chemical, and mechanical properties while generating a small quantity of heat.

(1) Aromatic anhydrides

Aromatic anhydrides are generally solid. They have been used in powder paints for powder molding, and their varnishes and solutions in liquid anhydrides have been used in insulating coating for condensers and casting.

(2) Alicyclic anhydrides

Alicyclic anhydrides are the most common curing agents for epoxy resin. Most of the generally used anhydrides fall into this category. Of these, the

principal curing agents are methyltetrahydro phthalic anhydride, tetrahydro phthalic anhydride, methyl nadic anhydride, hexahydro phthalic anhydride, and methylhexahydro phthalic anhydride.

(3) Aliphatic anhydrides

Polycarboxylic anhydrides, which are formed by the dehydration condensation reaction between aliphatic dibasic-acid molecules, exhibit excellent flexibility and thermal shock resistance, and have been used alone or in combination with other anhydrides in powder paints and casting resin curing agents.

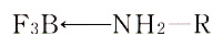
When anhydrides are used as curing agents, curing accelerators are normally used. Many types of accelerators, such as tertiary amine, boric-acid ester, Lewis acid, organic metal compounds, organic metal salts, and imidazole, have been studied.

7. Latent curing agents

Latent curing agents are mixtures of curing agents with epoxy resin that can be stably stored at room temperature, and rapidly cure by heat, light, pressure, and others.

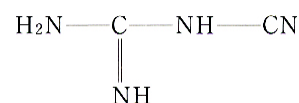
7-1. Boron trifluoride-amine complex (Three Bond 2285B, 2287, 2287B)

Lewis acids such as BF_3 , ZnCl_2 , SnCl_4 , FeCl_3 , and AlCl_3 have been known as cationic polymerization catalysts. Unlike anion polymerization by Lewis bases (e.g., tertiary amine), Lewis acids work as polymerization catalysts not only for DGEBA-type resins but also for linear and alicyclic epoxy resins. The Lewis acids vigorously react with resins at room temperature, and their pot life is 30 seconds or less. Therefore, they are normally used in the form of complexes with amines. The representative Lewis acid is boron trifluoride-amine complex, which has the structure shown below. The complexes have different properties (e.g., melting point, reactivity) according to the type of amine.



The BF_3 -amine complex is a catalyzed curing agent, and thus it is added to resins in small amounts (1% to 5%). The cured resins have a high HDT (150°C to 170°C); in particular, the cured substances of some novolac-type epoxy resins have a HDT of 230°C, and feature excellent electrical properties, but their chemical resistance is relatively weak. Due to the latency and heat resistance of the cured resins, the complexes have been used in electrical insulating laminates and carbon fiber reinforced plastics (CFRP).

7-2. Dicyandiamide (Three Bond 2220 to 2227)



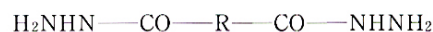
Dicyandiamide (DICY) is a representative latent curing agent that forms crystals having a high melting point of 207°C to 210°C. DICY has a pot

life of 24 hours when it is dissolved in epoxy resin using a solvent or the like, but it is normally used in the form of fine powder dispersed in the resin, which has a very long pot life of 6 to 12 months. DICY cures at high temperatures of 160°C to 180°C in 20 to 60 minutes, and generates a large quantity of heat during curing. Thus, it has used only in thin films such as paints, adhesives, and laminates. The cured resins have good adhesiveness and are less prone to staining. DICY has frequently been used in one-part adhesives, powder paints, and pre-pregs.

Although DICY has good latency, its curing temperature is so high and its curing time so long that it requires an accelerator. Commonly used basic compounds include tertiary amine, imidazole, and aromatic amine.

7-3. Organic-acid hydrazide

Organic-acid hydrazide, a powder that has a high melting point and is synthesized from carboxylate ester and hydrazine, is dispersed in epoxy resin to work as a latent curing agent.

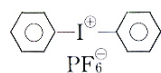


Normally, organic-acid hydrazide has a pot life of 4 to 6 months, and cures at ca. 150°C in 1 to 2 hours. Organic-acid hydrazide cures at lower temperatures and has better water resistance and adhesiveness than DICY, and has been used in powder paints and one-part adhesives.

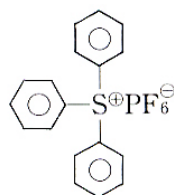
8. Light-curing and ultraviolet-curing agents (Three Bond 3101) —●

Light-curing and ultraviolet (UV)-curing agents are stable in epoxy resin, and are decomposed by exposure to light or UV to cure the resin. The agents are regarded as a type of latent curing agent, and are attracting attention as a potential ingredient of non-polluting paints and printing inks, in which the agents will be dissolved in liquid resins using no solvent.

The representative ultraviolet (UV) curing agents are the following two in the form of onium salt:

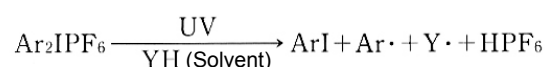


Diphenyliodonium hexafluorophosphate



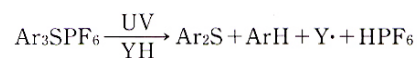
Triphenylsulfonium hexafluorophosphate

In 1975, Crivello used a diaryl iodonium salt for ring-opening polymerization of epoxy resin.



Above, the cationic polymerization of epoxy resin using HPF₆ is shown.

Crivello also found that triaryl sulfonium salt works as a light-curing agent for epoxy resin. It cures rapidly, particularly in the case of alicyclic epoxy resin.



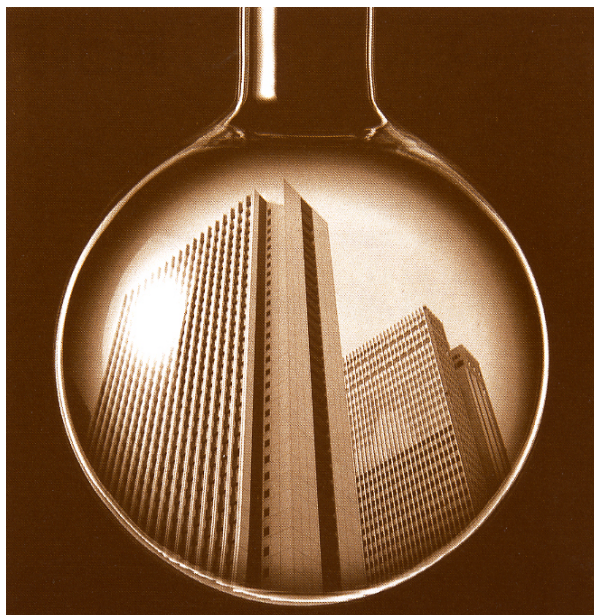
The light-curing and UV-curing of epoxy resins do not involve enzyme inhibitions that may affect the radical polymerization of photosensitive resins, and the cured epoxy resin has better properties and adhesiveness than other resins.

Conclusion

In this issue, we have reviewed various curing agents for epoxy resin. The most suitable curing agent must be selected according to the use conditions, application, workability, and other factors. Only the curing agents for epoxy resin are discussed in this issue; in practical use, however, they are mixed with diluents, coupling agents, fillers, and other agents to improve the curing properties and workability. As closing, I would hope

that epoxy resins are widely used in many fields.

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