Chapter

Epoxy Adhesives

Chunfu Chen, Bin Li, Masao Kanari and Daoqiang Lu

Abstract

Epoxy adhesives are primarily composed of epoxy resin and curing agent. Epoxy adhesives are supplied in both one-component package and two-component package depending on curing agent used and curing method applied. Two-component epoxy adhesives are prepared by packing epoxy composition and curing agent composition separately. They cure soon after mixing the two components together. Almost all room temperature cure epoxy adhesives are supplied in two-component package. One-component epoxy adhesives are prepared and supplied by mixing all formulated components in advance including epoxy resin and curing agent. One-component epoxy adhesives usually need cure at elevated temperature and store at low temperature in a refrigerator or even freezer. Epoxy adhesives have been widely used as typical reactive adhesives for various applications ranging from general industry, construction, electronics assembly, automobile production to aerospace market. Typical room temperature cure epoxy adhesives, thermal cure epoxy adhesives and UV cure epoxy adhesives are introduced in detail.

Keywords: epoxy adhesive, one-component, two-component, room temperature cure, thermal cure, UV cure, latent curing agent

1. Introduction

Epoxy adhesive was first invented in 1936 by Dr. Pierre Castan for dental application via curing bisphenol A epoxy resin with phthalic anhydride. Commercial supply of epoxy adhesives was started in late 1940s in Europe and USA. Various epoxy adhesives have been developed and commercialized since then and widely used as typical reactive adhesives for various structural bonding applications ranging from general industry, construction, electronics assembly, automobile production to aerospace market [1–11]. Their typical application area and examples as well as their supply type, curing method are summarized in **Table 1**. Major global suppliers for epoxy adhesives are Henkel AG & Co. KGaA, H.B. Fuller Company, 3M, Huntsman Corporation, Sika Corporation, Arkema Corporation, Cemendine Co., Ltd., Three-Bond Co., ltd., Huitian Adhesive, etc.

Epoxy adhesives show good adhesion on various substrates and are suitable to bond metals, glass, concrete, ceramics, wood and many plastics. Curing shrinkage is very low. Cured epoxy resin possesses strong and rigid cross-linked chemical structure suitable for structural bonding applications. By combination of various epoxy resins and different curing agents, a number of epoxy adhesives have been commercialized for different applications. On the other hand, room temperature and thermal cure epoxy adhesives need relatively long cure time. Most cured epoxy adhesives are very rigid and are not suitable for bonding flexible substrates.

Application area	Application examples	Package type	Cure method
Industrial	Structural bonding	One component; Two components	R.T. cure Thermal cure
Construction	Concrete repairing Anchor bolt fixture	Two components	R.T. cure
Automotive	Structural bonding Hemming adhesion	One component; Two components	Thermal cure
Aerospace	Metal, honeycomb & composite bonding, repairing	One component; Two components	Thermal cure
Electronics	Electrically conductive Display assembly Image sensor assembly Underfills Medical bonding	One component; Two components	Thermal cure UV cure R.T. cure
Others	Sports tools Consumer applications	Two components	R.T. cure

Table 1.
Typical applications of epoxy adhesives.

In selection and use of epoxy adhesives, cautions need to be paid on their pot life, cure condition, cure method, physical properties of un-cure and cured resin as well as adhesion performance.

2. Epoxy adhesive chemistry

Epoxy adhesives are primarily composed of epoxy resin and curing agent. Filler, toughener, plasticizer and other additives such as silane coupling agent, deformer and colorant, etc., can be formulated as needed. Common compositions and their main role of epoxy adhesives are illustrated in **Table 2**.

Epoxy resins are mainly synthesized from reaction of active hydrogen in phenols, alcohols, amines and acids with epichorohydrin, abbreviated normally as ECH at certain well controlled conditions. Epoxy resin can be also prepared by oxidation of olefin with peroxide as in the case of preparation of cycloaliphatic epoxy resins. Main commercial epoxy resins, their preparation and key features are shown in **Table 3**. Bisphenol A diglycidyl ether, often called as bisphenol A type epoxy resin, is the first commercialized and still most widely used epoxy resin. Synthesis of DGEBA is illustrated in **Figure 1** [12]. In volume base, it is estimated that over 75% of epoxy resin used in industry is this type. **Figure 2** illustrates chemical structure and key features of various functional groups for bisphenol A diglycidyl ether [13], the most common epoxy resin used in epoxy adhesives.

Epoxide group is chemically very active. Epoxy resin can react with active hydrogen almost equivalently via polyaddition mechanism with polyamines, mercaptan compounds, phenols and anhydrates to become cross-linked strong thermoset polymers. Epoxy resin can also polymerize homogeneously via anionic polymerization mechanism by initiating of Lewis bases such as tertiary amines or imidazole compounds. It can also polymerize via cationic polymerization mechanism via initiating of Lewis acid such as Boron trifluoride amine complex or strong acid such as onium salts, iodonium salts. **Table 4** lists typical curing agent, initiator used in epoxy adhesives. By combination of suitable epoxy resin with curing agent, epoxy adhesive is designed for various substrate bonding in different applications. It is supplied in both two-component and

Constituent	Ingredient	Main role
Primary	Epoxy resin, reactive diluent	Adhesive base
	Curing agent/catalyst, accelerator	Curability
Modifying	Filler	Property modification
	Toughener	Toughening
	Plasticizer	Flexibility
Additive	Coupling agent	Adhesion
	Colorant	Color

Table 2.
Epoxy adhesive compositions.

Epoxy resin type	Preparation from	Key features
Glycidyl ether of		
Bisphenol A Bisphenol F Novalac	Bisphenol A and ECH Bisphenol F and ECH Novalac with ECH	Standard epoxy resin Low viscosity Multi-functional
Glycidyl ester	Carboxylic acids and ECH	Mainly for anhydrate cure
Glycidyl amine	Amines and ECH	Multi-functional
Cycloaliphatic	Oxidation of olefin by peroxide	Cationic cure

Table 3.
Commercial epoxy resins.

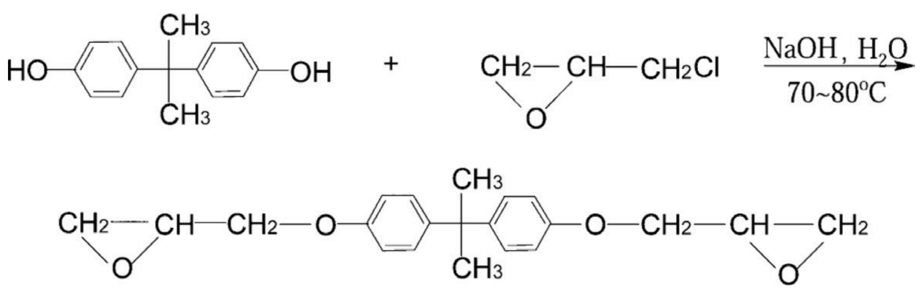

Figure 1.
Synthesis of DGEBA (diglycidyl ether of bisphenol A).

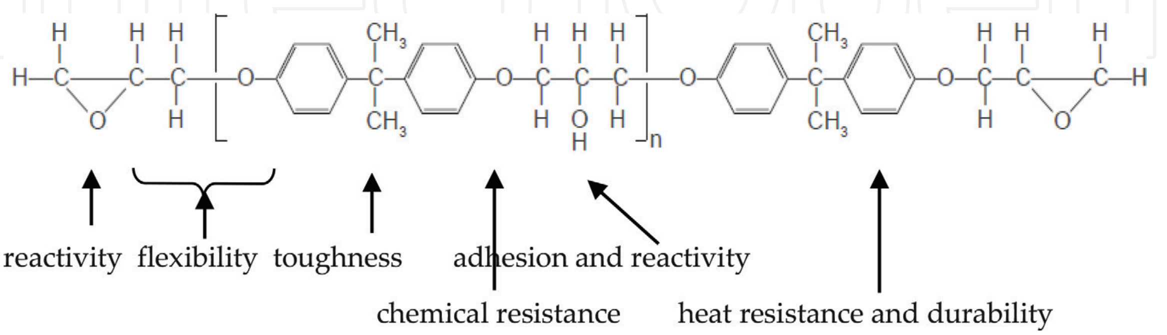

Figure 2.
Chemical structure and key features of DGEBA.

one-component package depending on the curing agent and curing method used. Two-component epoxy adhesive is prepared by packing epoxy composition and curing agent composition separately before use. It will cure soon after mixing based on designed mixing ratio. Almost all room temperature cure epoxy adhesives

Polymerization mechanism	Curing agent, initiator
Polyaddition	Polyamines Modified polyamines Mercaptans Phenols Anhydrates Dicyandiamide
Anionic	Tertiary amines Imidazole compounds
Cationic	Boron trifluoride monoethylamine Onium salts Iodonium salts

Table 4.
Epoxy curing agents.

are supplied in two-component package. Epoxy adhesives can be formulated in one-component package where all components including epoxy resin and curing agent has been mixed in advance. One-component epoxy adhesives usually use elevated temperature cure and need to be stored at low temperature conditions in a refrigerator or even freezer for long shelf life.

3. Room temperature cure epoxy adhesives

Room temperature cure epoxy adhesives are normally prepared and supplied in two-component package with epoxy resin component parked in one resin part and curing agent packed as the other hardener part. By mixing these two parts together, epoxy resin will react with curing agent quickly at room temperature conditions to become cross-linked strong thermoset structure that can bond adhesion substrates tightly. By use of different type of curing agents, pot life and cure time can be designed as needed.

3.1 Fast room temperature cure epoxy adhesives

Mercaptan compounds are usually selected as curing agent for fast room temperature curable epoxy adhesive because its reaction with epoxy resin is very fast in the existence of small amount of basic chemicals such as tertiary amine or imidazole as accelerator. As shown in **Figure 3**, epoxy resin reacts with mercaptan group equivalently via polyaddition reaction mechanism [14]. Fixture time can be <30 minutes or even 15 minutes at room temperature. Full cure time will need 24 hours. Precautions need to be paid on its very short work life, <10 minutes or even 5 minutes. Commercial fast cure epoxy adhesives supplied by Henkel AG & Co. KGaA and their typical properties are illustrated in **Table 5** [15].

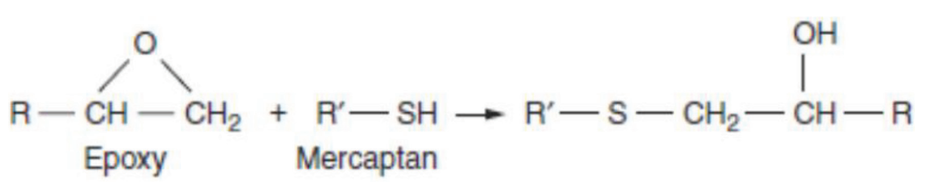

Figure 3.
Polyaddition reaction of epoxy resin with mercaptan.

Product	LOCTITE EA E-05MR	LOCTITE EA E-00NS
Color	clear	translucent
Viscosity, mPas/25°C	25,000	100,000
Mix ratio	1:1	1:1
Work life, minutes	5	3
Fixture time, minutes	15	10
Room temperature cure time, hours	24	24
Shear strength, psi on steel	3360	1600

Table 5.
Fast room temperature cure epoxy adhesives.

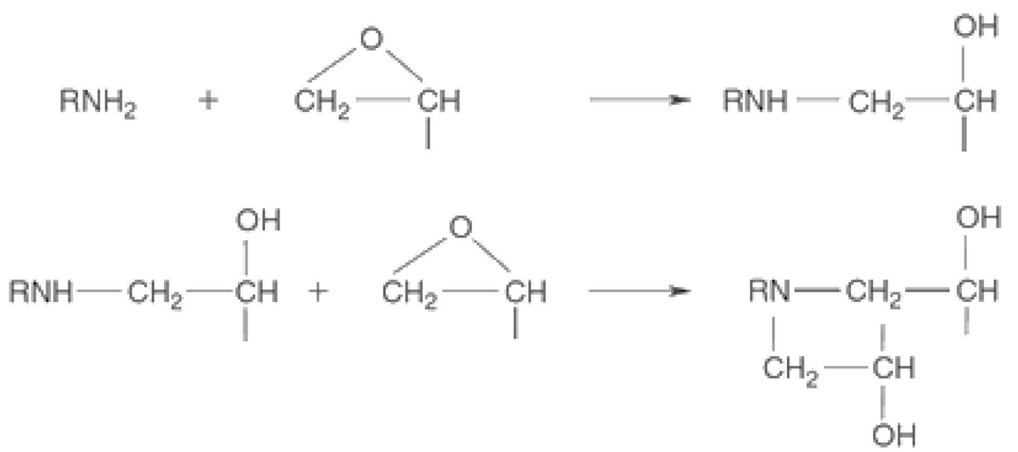

Figure 4.
Polyaddition reaction between epoxy resin and amine.

Product	LOCTITE EA E-20HP	LOCTITE EA E-120HP
Color	Off-White	Amber
Viscosity, mPas/25°C	30,000	30,000
Mix ratio	2:1	2:1
Work life, minutes	20	120
Fixture time, minutes	120	>180
Room temperature cure time, hours	24	24
Shear strength, psi on steel	3270	4300

Table 6.
Room temperature cure epoxy adhesives.

3.2 Room temperature cure epoxy adhesives

Aliphatic polyamines are most commonly used curing agents in epoxy resin technology. A number of modified polyamine type curing agents with adjustment on curability, handling or other physical properties for easy use have been commercialized in the market by curing agent suppliers. As shown in **Figure 4**, active hydrogen of primary and secondary amine reacts equivalently with epoxide via polyaddition mechanism [16]. Fixture time and work life can be adjusted by combination with suitable curing agent. **Table 6** shows commercial room temperature epoxy adhesives supplied by Henkel AG & Co. KGaA and their properties [17].

4. Thermal cure epoxy adhesives

Thermal cure epoxy adhesives are prepared and supplied in both one-component and two-component packages depending mainly on curing agent type used. Compared to room temperature cure type, thermal cure two-component epoxy adhesives usually have higher glass transition temperature that is suitable for high temperature resistance applications. One-component epoxy adhesives do not need pre-mixing in use and thus can be handled much easily. Many new one-component epoxy adhesives have been commercialized and become more and more important in recent years.

4.1 Two-component thermal cure epoxy adhesives

When use cycloaliphatic amine or aromatic amine as curing agent, post thermal cure process is usually required to achieve full cure as their reactivity, especially aromatic amines and secondary amine in cycloaliphatic amine, with epoxide is much lower with compared to aliphatic amines applicable for room temperature cure. Chemical structure of commonly used cycloaliphatic amine IPDA (isophorone diamine) and aromatic amine DDM (methylene dianiline) is shown in **Figure 5** [18, 19]. Thermal cure epoxy adhesives have much stronger and rigid structure and normally possess higher glass transition temperature with compared to room temperature cure epoxy adhesives mainly based on aliphatic amines or mercaptans. Two-component thermal cure epoxy adhesives are mainly used for higher temperature resistance required applications such as automobile production and aerospace market.

4.2 One-component thermal cure epoxy adhesives

One-component epoxy adhesives do not require pre-mix before use since all components have been mixed together and there is no concern on insufficient mixing problem as often occurred in two-component use. Pot life of one-component epoxy adhesives is usually long and one-component adhesives are thus suitable for automatic dispensing systems. Compared to two-component type, one-component epoxy adhesives can be handled much easily. On the other hand, one-component epoxy adhesives usually need cure at higher temperature because of long enough room temperature stability needed for adhesive preparation and storage. Most one-component epoxy adhesives require storage condition at lower temperatures in a refrigerator or even freezer.

Recently one-component thermal cure epoxy adhesives have become more and more important especially in electronics assembly and automotive production where high production efficiency is required. With selection of suitable latent curing agents, a number of one-component epoxy adhesives have been developed and commercialized by epoxy adhesive suppliers for various applications. Typical commercial latent curing agents are summarized in **Table 7**.

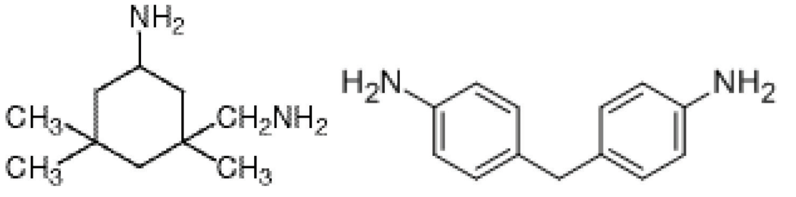

Figure 5.
Chemical structure of IPDA and DDM.

Latency mechanism	Latent curing agent	Curing agent state	Typical curing temperature °C
Chemical block and physical separation	DICY	Solid	≥150
	Dihydrazines		≥120
Physical separation	Modified imidazoles	Fine powder	≥80
	Modified polyamine		≥80
Chemical block	Onium salts	Solid	≥80
	Amine-BF3 complex	Liquid	≥130

Table 7.
Typical commercial latent curing agents.

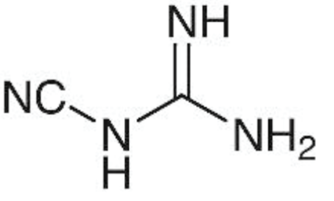

Figure 6.
Chemical structure of DICY.

DICY (dicyandiamide), chemical structure shown in **Figure 6** [20], is the oldest and widely used latent curing agent for epoxy resin technology. It is a solid chemical with a melting point at 208°C. DICY formulated epoxy composition is very stable, up to 6 months at room temperature. Latency mechanism is a combination of physically separation and chemically blocking with epoxide group. DICY cured epoxy resin shows high adhesion and possesses high glass transition temperature especially suitable for high performance required applications such as vehicle parts bonding in automobile production. Cure temperature of DICY alone with epoxy resin normally needs at least 150°C to achieve full cure. By addition small amount of accelerator such as modified urea compounds and imidazole compound, cure temperature can be further lowered to 120°C [21].

In recent years, new type latent curing agents have been developed and commercialized by several curing agent suppliers [22–26]. These latent curing agents are supplied as fine powder with average particle size well controlled in a few microns or premix of fine powder latent curing agent in epoxy resin. They are manufactured by grinding specially synthesized modified polyamine or imidazole solid with a softening point from 80 to 150°C. Latency mechanism is mainly physically separation between curing agent and epoxide. Curing temperature has been be lowered to as low as 80°C and its formulated epoxy composition can be still quite stable at room temperature. Many one-component epoxy adhesives commercialized recently are based on these new type latent curing agents because of their lower temperature curability suitable for use in bonding heat sensitive substrates such as plastics. By combination of small amount of liquid phenol compound with latent curing agent, it has been found that cure time of one-component epoxy adhesives can be shortened significantly [27, 28].

5. UV cure epoxy adhesives

Ultra-violet light (UV) curable epoxy adhesives can be cured quickly and have been very successfully used in several new electronics assembly and general bonding

Adhesive type	UV acrylate	UV cationic epoxy	Hybrid thermal cure epoxy
Main compositions	Acrylate	Epoxy resin	Acrylate
	Photoinitiator	Cationic photoinitiator	Photoinitiator
			Epoxy resin
			Curing agent
Polymerization			
UV cure	Radical	Cationic	Radical
Thermal cure	N.A.	Cationic	Polyaddition, anionic
Key features			
Oxygen inhibition	Yes	Yes	Partially
Alkali inhibition	No	Medium	No
UV curability	High	Preferred	High
Post thermal cure	No need	Partially	Need
Shadow cure	No	Low	Yes
Cure shrinkage	High	Good	Low
Adhesion	Moderate		Good

Table 8.
Comparison of UV acrylate, cationic epoxy and hybrid epoxy adhesives.

applications such as image sensor module assembly, display panel and module assembly where fast production speed and high adhesion performance are required. Various UV cationic epoxy adhesive and UV acrylate hybrid thermal cure epoxy adhesives have been commercialized in recent years. As compared in **Table 8**, UV cure epoxy adhesives have no oxygen inhibition issue, low curing shrinkage and show better adhesion with compared to common UV acrylate adhesives.

5.1 UV cationic epoxy adhesives

UV cationic epoxy adhesives are primarily composed of epoxy resin and cationic photo-initiator [29–31]. Cycloaliphatic type epoxy resins are usually selected for UV cationic epoxy adhesives because of faster cationic polymerization rate than that of normal bisphenol A diglycidyl ether type epoxy resin. As illustrated in **Figure 7** [32], cationic photoinitiator formulated in UV epoxy adhesives absorbs UV energy to generate strong acid that will react with epoxy to produce cationic which can initiate homo-polymerization of epoxy resin. Compared to common acrylate based UV adhesives, UV cationic epoxy adhesives have lower cure shrinkage because of epoxy structure and have no surface cure issue resulted from oxygen inhibition to free radical polymerization since they cure via cationic polymerization. On the other hand, UV cationic epoxy adhesives are not suitable for bonding basic substrates which terminate cationic polymerization. UV cationic epoxy adhesives will need some longer cure time. In real use, a post thermal cure of UV cationic epoxy adhesives after the UV radiation is commonly used for full cure to assure satisfactory adhesion performance.

UV cationic epoxy adhesives have been commercialized and used in optical parts bonding, camera module sensor packaging and OLED panel assembly applications [33–37]. The authors have found that adhesion reliability performance of UV cationic epoxy adhesives can be much improved by combination use of cationic photo initiator with thermal cationic initiator [38].

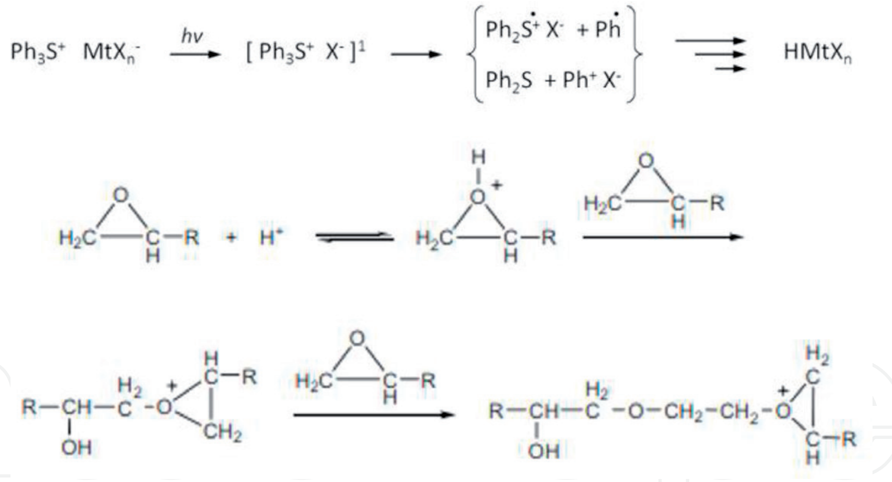

Figure 7.
UV cationic polymerization of epoxy adhesives.

5.2 UV hybrid epoxy adhesives

Most widely used UV cure adhesives are acrylate compositions [39–41]. Acrylate based UV cure adhesives are mainly composed of acrylate monomer, acrylate oligomer and radical photo-initiator. Acrylate based UV cure adhesives can be cured instantly, within seconds. Limitations of UV cure acrylate adhesives are surface cure issue, shadow cure problem, high cure shrinkage and poor humidity reliability. By combination of UV acrylate composition with thermal cure epoxy composition, UV and thermal cure hybrid epoxy adhesives have been developed and commercialized for over two decades [42–44]. Acrylate monomer, epoxy resin, photo-initiator and epoxy curing agent are primarily formulated in the UV and thermal cure hybrid adhesives. UV hybrid epoxy adhesives combine advantages from both UV acrylate proportion and thermal cure epoxy part. Adhesion reliability performance could be much improved by introduction of epoxy composition with compared to normal UV acrylate adhesives. In the meantime, production efficiency could be much improved by shortening the fixture time to seconds via UV radiation with compared to at least dozens of minutes needed for thermal cure epoxy adhesives. Surface cure issue, shadow cure issue and cure shrinkage problem of acrylate UV adhesives could also be improved to certain degree because of lower contents of free radical curable acrylate compositions. In some cases, thermal initiator such as peroxide is also formulated in the hybrid adhesive to assure curing remained acrylate compositions after UV radiation or those at shadow areas where UV light cannot reach.

Successful development and industrialization of so called ODF (One Drop Fill) process for large size LCD (liquid crystal panel) panel production was one important technology revolution in early 2000s that have made a big impact on our modern life. Development and commercialization of LCD ODF main sealant, an UV hybrid epoxy adhesive, played a key role in its mass production success [45–47]. LCD ODF main sealant is an adhesive material that is used to bond two glass substrates and seal liquid crystal material between them. It is a UV hybrid epoxy adhesive, typically composed of acrylate monomer, photo-initiator, partially acrylate epoxy resin and latent curing agent. Main steps for the adhesive use in this process are: (1) dispensing LCD main sealant; (2) dropping off liquid crystal materials into each cell; (3) alignment and assembly; (4) UV cure the sealant; and (5) thermal cure the sealant. The author has invented initiator free UV hybrid thermal cure epoxy adhesives by combination with bismaleimides that shows much better compatibility with liquid crystal material and high performance [48–50].

6. Summary

Epoxy adhesives show very good adhesion to various substrates and are the most important structural adhesives. Epoxy adhesives can be cured at room temperature condition, at elevated temperature condition or via UV light radiation mainly depending on curing agent type formulated. Lots of epoxy adhesives, either supplied in one-component or two-component package, have been commercialized and widely used for bonding metals, concrete, glass, ceramics, concrete, many plastics, wood, etc. in various industrial production and applications.

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