

A Review on Synthesis and Application of Epoxy Resins

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ABSTRACT:The simplicity of preparing and execution of cured items have made epoxy resins as material of decision in advanced composites which are fiber reinforced. Nonetheless, the epoxy resins are fragile in nature because of high cross-interface thickness. An expansion in molecular size of curing agent may prompt a decrease in cross-interface thickness. Flame resistance and thermal stability are another restriction of the epoxy resins. Nonetheless, the epoxy resins are fragile in nature because of high cross-interface thickness. An expansion in molecular size of curing agent may prompt a decrease in cross-interface thickness. Flame resistance and thermal stability are another restriction of the epoxy resins. Epoxy resins have been commonly used for coatings, electronic materials, adhesives and matrices for fibre-reinforced composites. The final properties of cured epoxy resins are influenced by the epoxy resin form, the healing agent, and the process of curing. In order to enhance the performance of epoxy resin in higher temperature, attention was focused on the development of novel curators containing amide group. This paper aims to study epoxy resins synthesis, process curing, and application.

KEYWORDS:Epoxy resins, Composites, Curing agent, Synthesis, Application

I. INTRODUCTION

Prileschajew discovered epoxy-resins in 1909[1]. Epoxy resins are classified as pre-polymers of low molecular weight, containing more than one group of epoxides in the form of Figure 1[2].

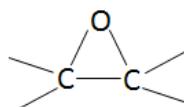


Figure 1: Structure of epoxy

Curing reactions make the epoxy resins thermosetting resins, when cured with wide range of curing agents. The properties of the epoxy resins depend on the specific combination of epoxy resin forms and the type of curing agents used [3]. Thanks to their outstanding mechanical properties, good adhesive to many substrates, and strong heat and chemical resistances, epoxy resins are currently intensively used in a wide variety of areas, where they serve as fibre-reinforced materials, adhesives, high-performance coatings, and encapsulating materials. The synthesis and curing method of epoxy resins is discussed in detail in this paper. The paper also addresses the preparation and application of epoxy-based composites.

II. SYNTHESIS OF EPOXY RESINS

Bisphenol-A epoxy resins

Reacting epichlorohydrin with bisphenol-A in the presence of a basic catalyst gives diglycidylether bisphenol-A (DGEBA). Figure 2 shows the chemical structure of DGEBA. The DGEBA resin properties are dependent on the number of repeated units. Liquid DGEBA has lower molecular weight whereas solids or more viscous liquid DGEBA has higher molecular weight [4].

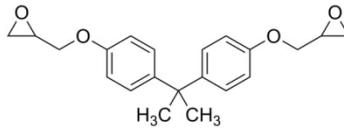


Figure 2: Structure of DGEBA

Yang et al.[5] synthesized epoxy resin with low viscosity by reaction of polyethylene glycol and DGEBA epoxy resin. Its epoxy Resin cured under UV light using a cationic photoinitiator, and the curing degree of epoxy resin within 40 s exceeded 90 percent. Czub[6] has been synthesized with high molecular weight epoxy resins from natural modified oils and bisphenol A, or epoxy resins made from bisphenol A. The results of the combination are that the liquids are extremely viscous. Liquefied Bamboo – bisphenol A copolymer epoxy resins in a two-step process was synthesized by Wu et al.[7]. Since the curing reaction is exothermic reaction, the curing of copolymer epoxy resin can be done at room temperature.

Cycloaliphatic epoxy resins

Reacting 3'-cyclohexenylmethyl 3-cyclohexenecarboxylate with peracetic acid gives 3',4'-epoxycyclohexyl- methyl 3,4-epoxycyclohexanecarboxylate, a cycloaliphatic epoxy resin (CAE). Chemical structure of CAE has given in Figure 3. This CAE has a saturated backbone and a backbone of aliphatic, contributing to its excellent UV stability; excellent electrical properties and good thermal stability. These characteristics are important for resins used in manufacturing of structural components which require high-temperature application in environment [8].

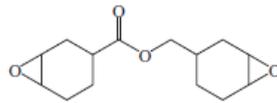


Figure 3: Chemical structure of CAE

1,3-bis[3-(4,5-epoxy-1,2,3,6-tetrahydrophthalimido) propyl] tetramethyldisiloxane (BISE) epoxy resin, an imide ring and siloxane-containing CAE. BISE on fully curing has a lesser glass transition temperature(Tg) compared with commercially available CAE and having a good thermal stability was synthesized by Tao et al.[9] A two-step reaction route of transparent cycloaliphatic epoxy-silicone for use in opto-electronic devices packaging was synthesized by Gao et al.[10]. Lower water absorption, higher thermal/UV resistance and better thermal stability are the properties exhibited by the cured cycloaliphatic epoxy-silicone resins.

III. TRIFUNCTIONAL EPOXY RESINS

Trimethylol propane-N-triglycidyl ether, shown in Figure 4, a trifunctional epoxy resin, can be prepared by reacting trimethylol propane and epichlorohydrin. This epoxy resin can be cured at low temperature since it has low-viscosity, non-crystalline and plastic material [11].

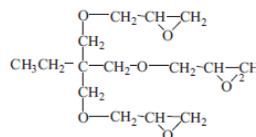


Figure 4: Structure of trimethylol propane-N-triglycidyl ether

Tetrafunctional epoxy resins

Reacting 1,3-diaminobenzene or 4,4'-aminodiphenyl methane with epichlorohydrin gives tetrafunctional epoxy resins. Figure 5 the chemical structures of tetrafunctional epoxy resins. As these epoxy resins have high cross linking densities and high epoxy functionality, they are used in places where high-temperature resistance is required. Good UV-blocking effect, excellent chemical resistance, good thermal stability and high modulus are the properties shown by the cured tetrafunctional epoxy resins [12].

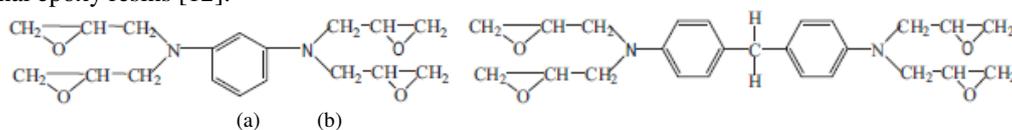


Figure 5: Chemical structures of tetrafunctional epoxy resins

A multi-functional epoxy resin was synthesized by Aouf et al.[13]. The cured epoxy resin showed a higher cross-linking density and a higher char yield than DGEBA cured under the same conditions.

Novolac epoxy resins

Phenolic novolacglycidyl ethers resins are derived from novolac epoxy resins are synthesized by reacting phenolic novolac resin with epichlorohydrin. The chemical structure of novolac epoxy resins is shown in Figure 6. Novolac epoxy resins have high cross-linking densities because of their multiple epoxide groups, thereby having an excellent chemical, solvent-resistance and thermal properties [14].

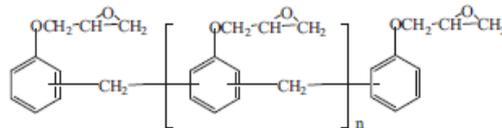


Figure 6: Chemical structure of novolac epoxy resins

Addition reaction of 9,10-dihydro-9-oxa-10-phosphaphenanthrene 10-oxide and novolac epoxy resin gives multifunctional phosphorus-containing epoxy resins. This multifunctional phosphorus-containing epoxy resin was synthesized by Lin et al. [15] exhibiting high thermal stability and high T_g, and achieved an impressive V-0 grade in the UL-94 vertical flammability test.

Biobased epoxy resins

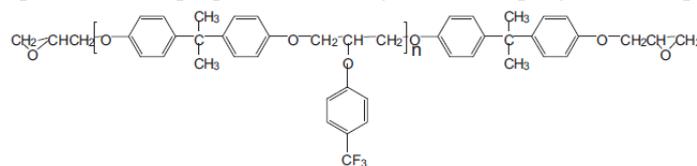
Proteins, starch, Carbohydrates, oils and fats, derived from renewable natural resources have attracted increasingly interest of their biodegradability and low cost. Vegetable oils contain complex multi-component mixtures of different triacylglycerols, i.e., esters of glycerol and fatty acids. Organic peracids or H₂O₂ with triglycerides having a large variety of unsaturations sites can be easily epoxidized. These epoxidized vegetable oil has excellent potential as renewable materials, inexpensive for industrial applications [16]. Epoxidized castor oil (ECO) and Epoxidized soybean oil (ESO) were synthesized by Park [17] and Thulasiraman et al. [18] by reacting glacial acetic acid/H₂O₂ with castor or soybean oil. The chemical structures of ESO and ECO are shown in Figure 7.



Figure 7: Chemical structures of ESO and ECO

Fluorine-containing epoxy system

Presence of fluorine in fluorinated monomers and oligomers to modify epoxy resins has imparted unique characteristics such as broad use temperature, low coefficient of friction, low water absorption, outstanding chemical resistance and low dielectric constant. Fluorine-containing polymers, which enhance the appeal for commercial use is used to fabricate entire products from speciality polymers [19]. CF₃-group-containing DGEBA epoxy resin was synthesized by Park et al. [20]. The chemical structure of CF₃-group-containing DGEBA epoxy is shown in Figure 8. A lower dielectric constant and mechanical properties are the properties shown by the cured epoxy resin compared to regular DGEBA.

Figure 8: Chemical structure of CF₃ group-containing DGEBA epoxy resin.

Fluorine containing epoxy resins was synthesized Park et al. [21]. The total surface energy of the tetrafunctional epoxy resin/fluorine-containing epoxy resins blends gradually decreased, whereas the water repellence of the blends increased with increase in the fluorine content. Through a four-step procedure a fluorinated epoxy resin, Tao et al. [22] synthesized 1,1-bis(4-glycidylesterphenyl)-1-(3'-trifluoromethylphenyl)-2,2,2-trifluoro-ethane (BGTF). Structure of BGTF has shown in Figure 9. Fully cured BGTF epoxy resin shows a good thermal stability with a glass transition temperature of 170°C -175°C and also improved dielectric properties compared to DGEBA epoxy resin.

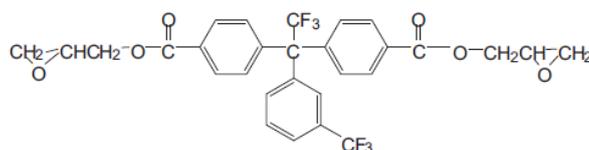


Figure 9: Chemical structure of BGTF.

Phosphorus-containing epoxy resins

Through flame inhibition in the gas phase and char enhancement in the condensed phase, phosphorous compounds could impart high flame-retardant character into epoxy resins. Phosphorous-containing epoxy resins generate less toxic gas and smoke than halogen-containing compounds. Using phosphorus-containing oxirane compounds, covalently bonded phosphorus into epoxy resins can be incorporated [23]. Environment-friendly electronic packaging materials containing phosphorus-containing epoxy resins was synthesized by Liu et al. [24]. Phosphorus-containing epoxy resins are shown in Figure 10. Epoxy resins were transparent with a high T_g of 227°C and high mechanical modulus after curing.

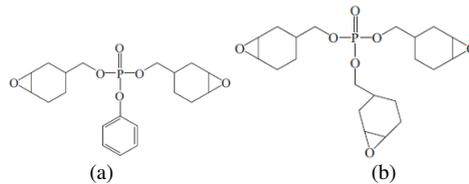


Figure 10: Chemical structures of phosphorus-containing epoxy resins

For the application of flame retardant materials, Wang et al. [25] synthesized phosphorus-containing epoxy resins. The phosphorus-containing epoxy resin and the nitrogen-containing curing agent showed synergistic effects and a great improvement in the flame retardant behaviour. High T_g , excellent flame retardancy and decomposition temperatures product was synthesised by Lie et al. [26] by reacting hexakis(4-hydroxyphenoxy)-cyclotriphosphazene, with DGEBA to form a new phosphazene-based epoxy resins.

Silicon-containing epoxy resins

As silicon has a less harmful effect on the environment than existing materials, it is regarded as an environment friendly flame retardant. Two approaches are used for synthesizing silicon-containing epoxy resins. One method utilizes the transesterification between alkoxy silane and glycidol, or the condensation of hydroxyl-terminated siloxanes with epoxy resins or epichlorohydrin. Another method is to introduce siloxanes into the epoxy compounds using hydrosilylation reactions. These epoxy resins display advantages of both epoxy resins and silicone resins [27]. Silicon-containing epoxy resins synthesized by Mercado et al. [28] is shown in Figure 11. A high limited oxygen index (LOI) value and moderate T_g are the properties of the cured silicon-containing epoxy resins.

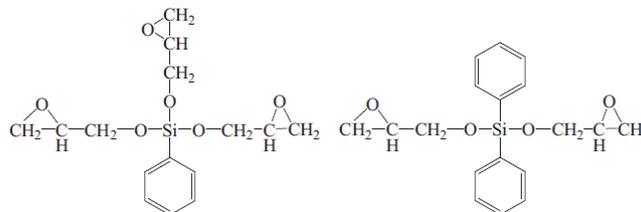


Figure 11: Chemical structures of silicon-containing epoxy resins

Two novel silicon-containing epoxy resins for electronics packaging applications is synthesized by Lie et al. [26]. Structures of these epoxy resins are shown in Figure 12. High T_g , good mechanical properties and good thermal stabilities are the properties of cured epoxy resins. Silicon-containing epoxy resins (DGEBA-Si) by addition of DGEBA with dichlorodiphenyl silane was synthesized by Park et al. [29]. Enhanced mechanical properties and lower T_g compared to pure DGEBA epoxy resin were the characteristics of the cured epoxy resin. Epoxy-terminated siloxane-containing resins were synthesized by Wang et al. [30] and the cured resin had high lap shear, good thermal stability and high tensile strength.

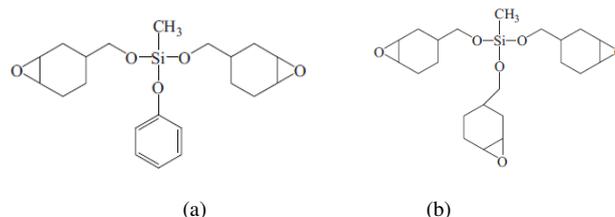


Figure 12: Chemical structures of silicon-containing epoxy resins

IV. CURING PROCESS

Promote or control the epoxy resin curing reaction, these are the properties of epoxy curing agents. Adding a curing agent enhances the epoxy resin curing. Molecular structure of the curing agents plays a major role in the cure kinetics and Tg [31]. Curing agents are classified based on their chemical compositions into catalytic curing agents.

Amine type curing agent

One of the curing agents for epoxy resins is the amine type curing agents. Amine type curing agents can be classified into three major categories: cycloaliphatic, aliphatic or aromatic amines. Epoxide rings react with amine type curing agents by nucleophilic addition. Chemical structures of 4,4'-diaminodiphenyl methane (DDM) and 4,4'-diaminodiphenyl sulfone (DDS) is shown in Figure 13 [32].

Alkali curing agent

Imidazoles and tertiary amines are categorized into alkali curing agents. In reactions between epoxy resins and other curing agents, imidazoles are used as primary accelerators. Imidazoles can be used as the sole curing agent [69]. Tertiary amines are also commonly used as accelerators for other epoxy resin curing agents. They are typically used in conjunction with amines, polyamides, amidoamines, mercaptans, polysulfides, and anhydrides to accelerate the epoxy-hardener reaction. Alkali curing agents offers a longer room temperature pot life [33].

Anhydride curing agent

Excellent electrical properties, good physical properties and good chemical resistance are the characteristics of anhydride curing agents. These curing agents possess a longer pot life. Amine catalyses the curing reaction of anhydride curing agents. The most common anhydrides are methyl tetrahydrophthalic anhydride (MTHPA), phthalic anhydride (PA), 3,4,5,6-tetrahydrophthalic anhydride (3,4,5,6-THPA), methyl hexahydrophthalic anhydride (MHHPA), hexahydrophthalic anhydride (HHPA), etc [34]. Figure 14 shows the chemical structures of PA and HHPA.

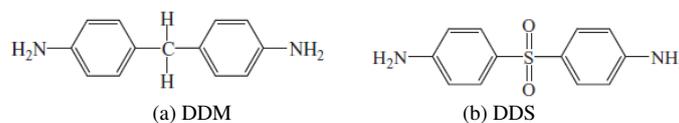


Figure 13: Chemical structures of DDM and DDS.

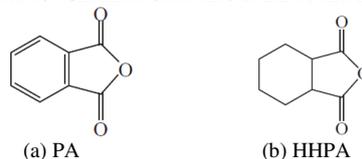


Figure 14: Chemical structures of PA and HHPA.

Catalytic curing agents

Catalytic curing agents are unreactive under basic conditions like, such as ambient temperature and internal lighting but they display activity with external simulation, such as heating or photo-irradiation. The curing proceeds by cationic polymerization initiated by the catalytic curing agent. Handling of epoxy resins and storage stability are enhanced by using catalytic curing agents. Benzylpyridinium, benzylationium, benzylammonium, and phosphonium salts are categorised into catalytic curing agents [35].

Curing systems

Epoxy resin curing systems comprise the chemical reactions of the epoxide groups in the epoxy resins with a curing agent to form a highly cross-linked, three-dimensional network [36].

Room-temperature curing

Epoxy resin curing that takes place at room temperature utilizes room-temperature curing agents, like alicyclic polyamines, aliphatic polyamines, improved aromatic amines and low molecular weight polyamide. Room temperature curing delivers a greater electrical resistance, lower Tg, greater impact resistance, higher flexibility and thermal shock resistance [37].

Heat curing

Largely, epoxy resins that are cured at higher temperatures employ dicyandiamide, resol resins, acid anhydrides, amino resins, aromatic polyamines, or hydrazides, as curing agents. The high-temperature curing system is commonly categorized into a pre-curing stage at low temperature and a post-curing stage at high temperature. Great chemical resistance, high T_g, great tensile strength and high heat resistance are the properties observed on curing epoxy resins at higher temperature. [38].

Photo-curing

Using bright light, electron beam irradiation infrared, ultraviolet light, epoxy resins are cured in the presence of a photoinitiator. Curing time can be reduced using photo-curing. In addition, it provides a more reliable and controlled process associated to the other curing processes [39].

V. EPOXY-BASED COMPOSITES

The highly cross-linked epoxy resins are strong and fragile and have relatively reduced resistance to crack initiation and growth, which minimise their use in many applications, such as structural materials. Various strengthening or toughening agents are used to improve their physical properties.

Thermoplastic-modified epoxy systems

Thermoplastic components like polysulfones (PSF), poly(acrylonitrile-co-butadi-ene-co-styrene) (ABS), poly(ether sulfones) (PES), poly(ether imides) (PEI), poly(ether ketone) (PEK), and polyimides (PI) are added into epoxy matrix to enhance the toughness of the epoxy resins. Thermoplastic modified epoxy systems are prepared by polymerization-induced phase separation of the thermoplastic modifier, which is primarily miscible in the epoxy resin but splits into phases during polymerization. Final properties of the systems are found by the resulting phase-separated morphology and effective improvement in toughness will be obtained only with a co-continuous phase or phase inversion structure [40].

Epoxy/inorganic composites

Epoxy resins are added with various inorganic particles to improve the fracture toughness. Increase in deformation and crack propagation resistance are seen on incorporating inorganic particles by toughening mechanisms, i.e., crack pinning/bowing, crack path deflection and particle bridging. Increase in hardness, fracture toughness and modulus is seen on increasing the inorganic particles [41].

Epoxy/carbon fiber composites

In a range of matrix materials, carbon fibers with high strength and modulus can be incorporated. At the point when stress/pressure is applied to a carbon fiber composite, the stress/pressure is moved from one carbon fiber to another through the matrix material. If a fiber-resin bond is frail, the stress will be transferred and fail or break the bonds between the resin matrix and fiber filaments. Sizing is a polymeric solution applied to enhance the adhesion. Carbon fibers are coated with sizing to enhance the adhesion with epoxy resin matrix [42]. Carbon fiber composites/ epoxy resins are utilized more widely for basic applications than other high performance composites due to their in general high strength properties and specific stiffness [43].

Epoxy/clay nanocomposites

Montmorillonite-based clay is a 2:1 layered silicate with sheets that transmit negative charges, which are balanced by the presence of interlayer cations (Na⁺ and Mg²⁺ ions). Agglomeration of the clay can be seen on fabrication of clay/polymer composites. Agglomeration of the clay results in degradation of the wear resistance and mechanical properties. The most well-known approach to defeat this trouble is to replace the inorganic Na⁺ cations on the clay surface with the essential organic cations, especially alkylammonium particles. When these surface-treated clays are brought into the host polymer matrix, two morphology types form: intercalated or exfoliated nanocomposite structures [44]. Clay/epoxy nanocomposites with lesser clay concentrations show improved thermal stability, mechanical strength, flammability and permeability compared to pristine epoxy matrices and conventional small scale/large scale composites. The superior performance of the clay/epoxy composites emerges from the ultra-high volume/interface ratio in the nanocomposites, resulting from the nanometer scale clay particles measurements [45].

Epoxy/carbon nanotube composites

Carbon nanotubes (CNTs) are built with length-to-diameter proportions that are fundamentally higher than that of some other materials, giving them phenomenal electronic, thermal and mechanical properties. Therefore, CNTs are useful fillers to improve the performance of the resulting composites. However, the low dispersibility and powerless interfacial interactions of CNTs inside epoxy networks have constrained their application around the area. Ultrasonic

scattering and high-shear mixing are examples of generally utilized mechanical techniques. Physical methods include the adsorption and additionally wrapping of polymers or surfactants on the CNT surfaces, and surfactants techniques comprise of covalent substance holding of polymer chains to the CNT surfaces, significantly improving interfacial associations among them and the epoxy network. A homogenous scattering of aligned CNTs with good chemical interactions with the epoxy resins can accomplish productive load transfer and provide a conductive pathway for electrons and phonons. CNT/epoxy composite present unique thermal, electrical and mechanical behaviours [46].

VI. APPLICATION FIELDS

Paints and coatings

Easy processing, mechanical and corrosion resistance, toughness, high safety, excellent solvent and chemical resistance, , low shrinkage on cure, and excellent adhesion to many substrates makes epoxy resins use as heavy-duty anticorrosion coatings. Metal cans and containers were coated with epoxy resins in order to prevent rusting, especially while packaging tomatoes, since is tomatoes are acidic foods. Epoxy resins are also used for decorative flooring applications and high performance such as terrazzo, chip, and coloured aggregate flooring [47].

Adhesives

Epoxy adhesives are categorised into a major part of the class of adhesives called as “structural adhesives”. Since epoxy adhesives have high strength bonds, therefore they are used in construction of aircraft, snowboards,, golf clubs, automobiles, bicycles, boats, skis. When used as adhesives in cryogenic engineering applications, it is necessary to optimize the epoxy shear strength at both cryogenic and room temperatures. Commercial epoxy adhesives are engineered for optimal toughness by incorporating phase-separated thermoplastics, rubber particles, or rigid inorganic particles into the matrix. Inorder to increase their strength and activate chemical bonding at the substrate/adhesive interface, the adhesives are cured at higher temperatures [48].

Industrial tooling

Epoxy systems are used in industrial tooling applications to produce molds, master models, laminates, castings, fixtures, and other industrial production aids. This “plastic tooling” replaces metal, wood, and other traditional materials, and generally improves the process efficiency while either lowering the overall cost or shortening the lead-time for many industrial processes. Fiber-reinforced epoxy composites have proven effective in repairing metallic components and tubular pipes. The composites also act as load-bearing units in hydrogen storage cylinders [49].

Aerospace industry

Low cost and high adhesive properties make epoxy resins are used extensively for structural adhesive application in aerospace industry. Epoxy resins reinforced with high strength glass, carbon, Kevlar, or boron fibers have the the most prominent potential for use in the aerospace industry [50].

Electronic materials

Epoxy resins are excellent electrical insulators and protect electrical components from short circuiting, dust, and moisture therefore they are employed in motors, generators, transformers, switchgear, bushings, and insulators. Electromagnetic interference shielding are used with the help of metal-filled polymers. Epoxy molding compounds (EMCs) are popularly used as encapsulation materials for semiconductor devices protect the integrated circuit devices from moisture, mobile-ion contaminants, and adverse environmental conditions such as temperature, radiation, humidity, and mechanical and physical damage [51]. Epoxy composites containing particulate fillers, such as fused silica, glass powder, and mineral silica have been used as substrate materials in electronic packaging applications [52].

Biomedical systems

Epoxy gums are generally utilized in biomedical applications. Collagen-based materials have been utilized in human clinical applications as wound dressings, vascular unions, and aortic heart valves [53]. Shape memory polymer foams have noteworthy potential for biomedical applications, for example, embolic sponges. Nanodiamond-epoxy derivatives have discovered extensive application in biomedical systems since they show a blend of extraordinary hardness, remarkable chemical idleness, low electrical and high thermal conductivities, wide optical transparency, and other remarkable properties [54].

VII. CONCLUSIONS

Different epoxy resins and curing agents have been evaluated in this paper. The properties of restored epoxy resins rely upon the kind of epoxy resins, curing agent, and curing process utilized. The strength of epoxy resins can be improved by consolidating thermoplastic parts, inorganics, carbon fibers, clay, and carbon nanotubes. Epoxy resins have a wide scope of utilization including coatings, aeronautic industry, electronic materials, and biomedical systems.

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