



The effect of finely-divided fillers on the adhesion strengths of epoxy-based adhesives



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ABSTRACT

It was aimed to investigate the effect of five different fillers on the adhesion strength of the metal parts used in different industries by using T-peel and lap-shear tests with respect to ASTM D 1876 and ASTM D 1002 respectively. EPIKOTE[®] 828 and DURATEK[®] epoxy resin types were used as adhesives with curing EPIKURE[®] 3090 modified polyamidoamine adduct and DURATEK[®] polyamide curing agents respectively. Curing temperatures were increased, while curing times were decreased. Five different fillers used are calcite with a mean particle diameter of 4.97 μm , talc extra with an average particle diameter of 4.91 μm , and barite extra with a mean particle diameter of 4.76 μm supplied by ESEN MIKRONIZE, calcite with an average particle diameter of 0.7 μm and coated calcite with a mean particle diameter of 0.9 μm provided by ERCIYES MIKRON. The effect of five different fillers on adhesion strength was investigated with choosing the best regression model fitted to data by using two different epoxy resin systems. The best values in shear and peel tests were attained with calcite having the particle diameter of 0.7 μm in both type of epoxy resins providing a good agreement between experimental and theoretical results.

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1. Introduction

A composite material is an engineering material that is consisted of two or more components that remain separate and distinct on a macroscopic level while existing a single material. Composite materials have a continuous matrix phase which binds a stronger reinforcement phase. The matrix binds and protects the reinforcement and gives the part form, while the reinforcement achieves desired structural and physical properties [1].

There are two types of classification for composite materials. They can be classified according to the matrix type and reinforcement form. There are also three types of matrices: organic matrix composites (OMCs), metal matrix composites (MMCs) and ceramic matrix composites (CMCs). OMCs contain polymer matrix composites (PMCs) and carbon matrix composites (CMCs) [1].

Filler is the most common type of reinforcement material used in PMCs and other types of composites. Operating temperatures have great importance on reinforcement as well as the size of the filler used to enhance bonding [1].

The matrix in a polymer matrix composite has three effects: transfer of stress between the filler particles, protect the surface

of the fillers from mechanical abrasion and provide a barrier against an adverse environment. The matrix material plays a significant role in certain loading cases and greatly influences the strength of a component. The matrix has a major influence in the interlaminar shear properties and in-plane shear properties. The interlaminar shear properties are important when a component is exposed to bending loads while the in-plane shear properties have a great role when a component is subjected to torsion. The matrix also provides lateral support, which contributes to a component's buckling properties and therefore compressive strength. The matrix has a little influence on the tensile properties of a component [1].

There are generally two types of polymer matrices containing thermoset and thermoplastic. Thermoset plastic is the most common matrix and includes over 80% of the matrices in reinforced plastics. The two most common types of thermoset plastic are polyester and epoxy [1].

There are three elements in an epoxy resin: the base resin, the hardener and the modifiers. The base resin describes certain properties of the final product such as operational temperature while the hardener defines the curing properties such as time and reaction initiation type (heat initiation or mixing initiation). Modifiers are added to produce specific physical and mechanical properties [1].

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Epoxy resins taking place in the family of thermosetting are used in many applications like composites, coatings, adhesives and encapsulating materials. Epoxy resins also belong to a family of molecules or oligomers having more than one epoxide group (oxirane). The chemical elected to react with these epoxides is known as the curing agent (or hardener), and it has active hydrogen attached to nitrogen, oxygen or sulfur. Many parameters affect the curing agent chosen and this selection influences the performance of the final epoxy structure as well [2].

A chemically modified amine epoxy curing agent provides flexibility in formulations ascending the performance without employing the disadvantages of traditional hardeners. Traditional aliphatic polyamines such as diethylenetriamine, triethylenetetramine and other aliphatic hydrocarbon-based amines have been extensively used as hardeners in ambient-cure-temperature epoxy resin systems. Cycloaliphatic diamines such as isophoronediamine, bis-*p*-aminocyclohexylmethane and 1, 2-diaminocyclohexane and aromatic diamines such as diaminodiphenylmethane (MDA) are also used as epoxy resin hardeners for both ambient and heat-cure epoxy resin systems [3].

Although epoxy resin and hardener [4] may be used in unmodified form for adhesive systems, most systems consist of modified components by incorporation of various additives to achieve specific effects. A lot of additives can be added to increase adhesion to difficult surfaces. Even though amidoamine hardeners have poor compatibility with respect to other hardeners especially in low temperature cure, addition of small quantities of imadazoline enhances adhesion to metals [5].

Epoxy resins provide excellent adhesion to a wide range of substrates such as metals, wood, glass and ceramics. This advantage makes them indispensable in adhesive applications where high strength is a prerequisite. Other advantages of them consist of negligible shrinkage during cure, good impact resistance, excellent chemical resistance except under the most severe acidic conditions, ability to bond nonporous substrates, endurance at elevated temperatures, high relative humidity [6] and great versatility [5]. Flexibility and toughness are significant properties of the epoxy adhesives as well.

The bonding of an adhesive to an adherent is the sum of the mechanical, physical and chemical forces affecting one another. Separation of these forces from one another is not possible, however, a difference can be made between chemical adhesion mechanism and the other adhesion mechanisms. Because, chemical bonds are strong enough to provide the bonding of the adhesive to the surface owing to the chemical reaction [7].

Joint design is one of the most significant elements in adhesive bonding. Joint geometry, adhesive selection, mechanical properties of adhesive and adherent, stress in the joint and manufacturing conditions are the main considerations in the design of bonded joints. Shear or peel stresses, often in combination can be applied in bonded joints [8].

Mechanical properties of adhesives were investigated by some researchers [9–11]. Studies made for numerical analysis of adhesive bonded joints were met [12–14].

Adhesive properties of epoxy resins were also explored [15–17]. Failure and fracture analysis of epoxy resins were studied [18–24]. Some experimental studies related to the epoxy resins were also made [25–31].

2. Experimental

2.1. Materials

EPIKOTE[®] resin 828, liquid epoxy resin produced from bisphenol A and epichlorohydrin and EPIKURE[®] curing agent 3090,

modified polyamidoamine adduct capable of curing epoxy resin at and below room temperature were obtained from Shell Chemical Company (TURKEY) [32,33]. Also, KLM 606A epoxy resin and KLM 606B polyamide curing agent were obtained from DURATEK[®] Company (TURKEY). Five different fillers were used. Calcite with a particle diameter of 4.97 μm , talc with having a particle diameter of 4.91 μm and barite with a particle diameter of 4.76 μm were supplied by ESEN MIKRONIZE (TURKEY), calcite having a particle diameter of 0.7 μm and calcite coated with the particle diameter of 0.9 μm were purchased by ERCIYES MIKRON (TURKEY). CAB-O-SIL TS-720 was obtained by CABOT. Low carbon alloy steel was used as adherent.

2.2. Surface preparation

The surfaces of adherents were prepared before application of adhesives. For this reason, two surface pretreatments were employed. First of all, steel specimens were cleaned with a liquid detergent solution, then, they were washed with hot water and dried in the oven. After then, the same degreasing operation was repeated once more. Finally, abrasion was applied to joint surfaces of adherents.

2.3. Preparation of samples

Ten different types of adhesives were prepared. Five adhesives were obtained by mixing DURATEK[®]KLM 606A epoxy resin and DURATEK[®]KLM 606B polyamide curing agent with a ratio of 1:1 by weight [34,35], adding CAB-O-SIL TS-720 rheology control agent. Preparation of five different adhesive samples were completed by using five different fillers (calcite obtained by ESEN MIKRONIZE with a mean particle diameter of 4.97 μm , talc extra purchased by ESEN MIKRONIZE with an average particle diameter of 4.91 μm , barite extra supplied by ESEN MIKRONIZE [36] with a mean particle diameter of 4.76 μm , calcite provided by ERCIYES MIKRON with an average particle diameter of 0.7 μm and coated calcite obtained by ERCIYES MIKRON [37] with a mean particle diameter of 0.9 μm). The adhesive samples were mixed by a mechanical mixer for five minutes according to the instructions given for DURATEK[®]KLM 606 epoxy resin and curing agent. The other five different adhesives were prepared with the addition of EPIKOTE[®] 828 epoxy resin, EPIKURE[®] polyamidoamine curing agent 3090 with a ratio of 1:1 by weight, using CAB-O-SIL TS-720 and five different fillers containing above specifications explained in detail. Those adhesives were also mixed by a mechanical stirrer for fifteen minutes approximately to insure the complete mixing. In this study, amount of CAB-O-SIL TS-720 used and content of five different fillers were kept as constant.

2.4. Peel test

After preparation of ten different adhesives, they were applied to the each surface of the steel adherents having the dimensions given in Fig. 1.

Adhesive thickness applied was kept as constant at 0.03 mm in all samples. Then, steel adherents were fitted in place. Next, they were cured at five different ovens with the temperature of 65 °C for 6 h, 80 °C for 4 h, 100 °C for 3 h, 120 °C for 2 h and 150 °C for 80 min. After then, joint samples were cooled to room temperature. Finally, peel tests of samples were performed with respect to the ASTM D 1876 by T-peel. T-peel adhesion was determined in terms of the force necessary to strip off the steel adherent from the other steel adherent with 180° angle at a peel rate of 12.7 mm/min by means of Shimadzu testing machine. Each result obtained was the average of five test results.

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