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Effects of coupling agents on the properties of epoxy-based electrically conductive adhesives

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Abstract

The study was carried out to investigate the effects of two different coupling agents on the properties of epoxy-based electrically conductive adhesives (ECAs). For the investigation, a novel mixture of solvent and diluted acid was designed to remove the lubricants on the surface of silver flakes fully and each of the following two commercially available coupling agents, a silane coupling agent and one titanate coupling agent, was used as an additive for the ECA. It was found that with the full removal of lubricants on the surface of silver flakes, the electrical conductivity is improved at the same weight filler loading. It was also found that the adding of coupling agents has different effects on the electrical, aging and lap shear properties of ECAs. According to this study, the use of silane coupling agent had a significant electrical conductivity improvement and lap shear strength increase before and after the aging test. But for the titanate coupling agent, the properties of the ECA have no significant change. Scanning electron microscopy studies revealed a strong adhesion/bonding between the filler and the matrix for coupling agent-added ECA.
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1. Introduction

Electrically conductive adhesives (ECAs), which consist of a polymer binder that provides mechanical strength, and conductive fillers, which offer electrical conduction, have gained much attention in the past decade, owing to their environmental and technical profits over traditional tin/lead (Sn/Pb) solders. ECAs provide a promising environmentally friendly solution for interconnections in electronic applications. Moreover, ECAs also offer several potential advantages over conventional solder interconnection technology, including finer pitch printing, lower temperature processing, and more flexible and simpler processing [1,2]. In addition, compared with lead-free solders, conductive adhesive systems exhibit greater flexibility, creep resistance and energy damping [3].

Epoxy-based materials have been widely used in engineering components because of their outstanding mechanical and thermal properties as well as processability. In ECAs, especially isotropically conductive adhesives (ICAs), the use of epoxies has been "the state-of-the-art" for a long time. This is due to the many beneficial properties of epoxies. They have low shrinkage, good adhesion and resistance to thermal and mechanical shocks. They also have good resistance to moisture, solvents and chemical attacks. The desirable properties may further be improved with a suitable choice of solvents, fillers, colorants, flame retardants, flexibilizers and cure accelerators [4]. Silver (Ag) flakes are widely used as fillers for ECAs.

To improve the adhesives' electrical and thermal conductivity, the amount of conducting filler particles may be increased. Experiments have, however, shown two results. One is that the maximum amount of silver

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particles in an epoxy matrix is approximately 30% by volume, and the second is that the adhesion of the adhesive is severely degraded. The amount of silver in ICAs is therefore typically between 70% and 80% by weight [4].

A lot of work has been done to improve the electrical and mechanical properties of ECAs in the past decade [5]. Some results also showed that the part removal of organic lubricants on the surface of silver flakes can promote the conductivity of ECAs [6,7], which play an important role for the performance of ICAs, including the dispersity of the Ag flakes in the adhesives and rheology of the adhesive formulation.

To modify properties of a highly filled polymer system, many additives have been developed, whose development is as much of an art as it is a science. Among these additives, coupling agents have gained more attention because of their special structures, which have two different functional groups, one that is attracted to the resin and the other that is attracted to the surface of the filler. There are many different proprietary alkylsilanes, organozirconates and organotitanates that have been developed for use with specific composites. Coupling agents are usually used for inorganic fillers-contained plastics to improve the adhesion between fillers and polymer, preferably via chemical bonds; their use improves the performance of reinforcements. There is extensive literature pertaining to the mechanical behavior of filled polymer, which focused attention on the influence of coupling agents on the physical and mechanical properties of inorganic filled composites [8–15]. Coupling agents are also adhesion promoters between substrate and adhesive, which can be applied directly to the substrate, similar to primers, or they can be mixed with the adhesive itself. When mixed with the adhesive, the coupling agents are capable of migrating to the interface and reacting with the substrate surface as the adhesive cures. When applied directly to the substrate, coupling agents are applied in a very thin coating that ideally is only one molecular layer thick [16]. When coupling agents are used in the design of electrically stable interfaces of 6061 T6 aluminum alloy surfaces and epoxy-based ECAs, it shows that besides their traditional use as surface modifiers for adhesion improvement, organo-silanes can act as corrosion inhibitors of aluminum surfaces to stabilize electrical performance [17].

In this paper, the influence of a silane coupling agent and a titanate coupling agent on the electrical and mechanical properties of epoxy-based ECAs was reported. For the investigation, a novel mixture of solvent and diluted acid was employed to remove the lubricant layer on the surface of silver flakes; the effects of lubricant layer were also studied.

2. Experimental

2.1. Material

The epoxy resin used in this study was the diglycidyl ether of bisphenol F (DGEBF, NPEL-170), lower in viscosity than standard bisphenol A resin, which was generously provided by Nan Ya Plastics Corporation, Taiwan. The silane coupling agent (KH-570, equivalent with A-174 of Union Carbide) and the titanate coupling agent (NDZ- 401, equivalent with KR-41B of Kenrich Petrochemicals, Inc.) are commercial products of Nanjing Shuguang Chemical Group Co., Ltd. The silver nitrate was provided by Hubei Xinying Noble Metal Co., Ltd. Other chemicals are commercial products and used without further purification.

2.2. Preparation of silver flakes with and without chemisorbed oleic acid

A process for preparing silver flakes comprised the following steps: (a) preparing an admixture of precipitated silver powders, which were prepared by a reduction process of the silver nitrate solution and the reducing agent hydrazine hydrate; the liquid milling medium used in this experiment was water, and about 1% by weight oleic acid, hydrophobic unsaturated; (b) bead-milling the admixture for a sufficient time; and (c) separating the liquid milling medium and beads from the resultant silver flakes particles. After production, the silver flakes have a thin layer organic lubricant, chemisorbed on the surfaces, labeled as S1. Some S1 silver flakes were treated with a novel mixture of alcohol and diluted sulfuric acid, and silver flakes with lubricants fully removed were obtained, labeled as S2. The difference between S1 and S2 is only that there are no lubricants on the surface of silver flakes S2.

2.3. Preparation of ECAs

Simplifying the effects of different parameters of the ECA composition, the ECA composition was simply composed of epoxy, curing agent and silver flakes fillers. Four types of ECA were designed: one is the mixture of polymer binder and silver flakes S1, denoted as A1; the polymer binder in the second composition is similar to A1, the silver flakes was S2, this ECA was denoted as A2; the main compositions of the third and fourth types of ECA are similar to A2, two coupling agents, the silane coupling agent (KH-570) and the titanate coupling agent (NDZ- 401) with 1% by weight of loading. Silver flakes were added to the third and fourth ECA, respectively, denoted as C1 and C2. The polymeric binders of the four ECAs are the same, which are made up of epoxy and hardener. To facilitate the mixing and dispensing of the conductive adhesive pastes,

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