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Influence of iron carbide filler in carbon matrix on the adhesive properties of acrylic pressure-sensitive adhesives

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ABSTRACT

Inorganic fillers can improve coating properties, such as scratch resistance and UV stability and can significantly enhance the fillers usability in coatings and realize new market opportunities. In the pressure-sensitive adhesive (PSA) technology the inorganic fillers are used to change the very important properties of pressure-sensitive adhesives, such as tack, peel adhesion and shear strength. In the current study, the above mentioned properties of synthesized acrylic PSA using iron carbide filler in carbon matrix were investigated. The acrylic PSA containing iron carbide filler (Fe_3C,C) was examined with SEM/EDX technique and the PSA adhesive and cohesive properties were examined by using a special strength machine according to AFERA tests. The conclusion is that the application of this kind of fillers allows the manufacturing of self-adhesive materials with moderate adhesiveness and very good removability.

1. Introduction

Acrylic pressure-sensitive adhesives are important materials due to their good adhesiveness and mechanical properties in combination with processability, compatibility with wide range of additives, such as dyes, resins, fillers and good thermal stability [1].

Filler technology represents a genuine technological advance for the widely understood coatings industry. Additives such as inorganic fillers are a small but indispensable part of coatings, paints, plastics, adhesives and pressure-sensitive adhesives (PSA). Most of the commonly used fillers act on surface or general interface properties. In adhesive application, many different types of interfaces exist and can be influenced by surface-active ingredients. For instance, wetting and dispersing additives stabilize the interface of solid materials dispersed in a continuous liquid phase [2–5].

Polymers containing diverse fillers are two-phase systems consisting of polymers loaded with high-surface area reinforcing fillers [6,7]. Such systems have attracted enormous interest from the materials community because of the theoretical promise at very low filler loadings. In addition, polymers containing inorganic fillers are compatible with conventional polymer processing, thus avoiding the costly layup required for the manufacturing of conventional fiber-reinforced composites [8]. Inorganic fillers, such as rods, cubes, prisms, tubes, fullerenes, cubic silsesquioxanes, dots, carbon blacks and silicium dioxide are available today in the market. They can be incorporated into polymer backbone to reduce costs, improve or regulate its properties [9].

For nonpermanent, the so-called removable, pressure-sensitive adhesives, the flow properties and cohesion of the adhesive as well as the anchorage of the adhesive to the face stock are critical. In an ideal case, if the bond to the substrate is nonpermanent, than a clean release from that substrate is encountered and the adhesive remains on the face material. Generally, a special balance between tack, peel adhesion and shear strength is required in order to ensure removability.

The application of iron carbide filler (Fe_3C,C) in carbon matrix is not known in fillers technology and is used in the present study.

Motivation of this work is provided by the influx of PSA layers designed to adhere to steel and other substrates such as aluminum or glass in the self-adhesive removable materials industry. These kinds of self-adhesive products consist of removable labels and sticky notes. Current evaluation of PSA behavior is accomplished by techniques such as tack, peel adhesion, shear strength and removability that were designed for these special important applications [10]. With these concepts in mind, we will examine the wettability of investigated inorganic fillers. We will explore the obtained iron carbide filler (Fe_3C,C) in carbon matrix in solvent-based synthesized acrylic pressure-sensitive adhesives (PSA), in fact in soft matrices. This paper is not an attempt to review the immense literature on filler composites. Rather, we focus on the relationship between wetability, morphology of

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reinforcing phase and the adhesiveness and mechanical properties of the resulting PSA containing tested iron carbide filler (Fe_3C,C) in carbon matrix.

In this work the first results of a systematic experimental study to characterize the effects in inorganic fillers on PSA–iron carbide interfacial properties were summarized.

2. Experimental

2.1. Synthesis and crosslinking of acrylic PSA

The solvent-borne acrylic PSA was synthesized in polymerization medium ethyl acetate at a temperature of 78 °C with 50 wt% polymer content using 97 wt% 2-ethylhexyl acrylate monomer and 3 wt% acrylic acid in the presence of 0.1 wt% radical starter 2,2′-azo-bis-diisobutyronitrile (AIBN). The monomer mixture was dosed into polymerization reactor in 2 h and the post-reaction time to reduce the residual monomers was 4 h. All components were available from BASF (Germany).

The synthesized acrylic PSA was stabilized with 30 wt% of isopropyl alcohol and crosslinked using 0.35 wt% according to polymer content of titanium acetyloacetonate (TiACA) available from Union Carbide.

2.2. Evaluation of the viscosity and residue monomers of synthesized acrylic PSA

The viscosity of the investigated solvent-borne acrylics pressure-sensitive adhesives was determined with a Rheomat RM 189 from Rheometric Scientific, with spindle no. 3 at 23 °C.

The amount of solid materials was found according to DIN EN 12092, and the residual monomers were measured with a gas chromatograph Unicam 610, J&W DB-1 column, FID detector and integrator Unicam 4815.

2.3. Preparation of iron carbide filler (Fe_3C,C) in carbon matrix

In carbonizing process the pure nano-crystalline iron recrystallizes quickly and forms polycrystalline–microcrystalline materials. For investigations fraction crystallite with a diameter of about 32 nm was selected. The tested nano-crystalline iron is characterized by specific surface 4.3 m^2/g and void ratio of 0.5.

Catalytic decomposition of methane was conducted in tubular reactor with thermogravimetric analysis of mass (Fig. 1). Samples of iron carbide were received by carbonizing of nano-crystalline iron using methane at a temperature of 650 °C. The carbonizing ratio, which is the mole number of carbon to mole number of iron, was 1.

The resulted iron carbide (Fe_3C) in carbon matrix was analyzed using an X-ray diffractometer (Fig. 2).

2.4. Modification of synthesized acrylic PSA through addition of iron carbide filler

The modification trials of synthesized acrylic PSA were conducted using prepared iron carbide (Fe_3C) in carbon matrix characterized by average particle size of 32 nm and specific surface of 4.3 m^2/g in amounts of 1.3; 2; 3; 4.5; 7 and 10 wt% with reference to acrylic polymer content. The mechanical incorporation of iron carbide into acrylic pressure-sensitive adhesives was achieved by mixing the mentioned filler with synthesized acrylic PSA using a dissolver at the very high mixing speed of 10,000 rpm for 10 min. No problems with the dispergation process of iron carbide into acrylic pressure-sensitive adhesives were observed.

2.5. Measurement of PSA properties containing iron carbide filler

The solvent-borne acrylic PSA containing iron carbide fillers are coated directly with 60 and 90 g/m^2 on a polyester film and dried for 10 min at 105 °C in a drying canal. The resulting PSA layers with iron carbide fillers are protected with silicon paper.

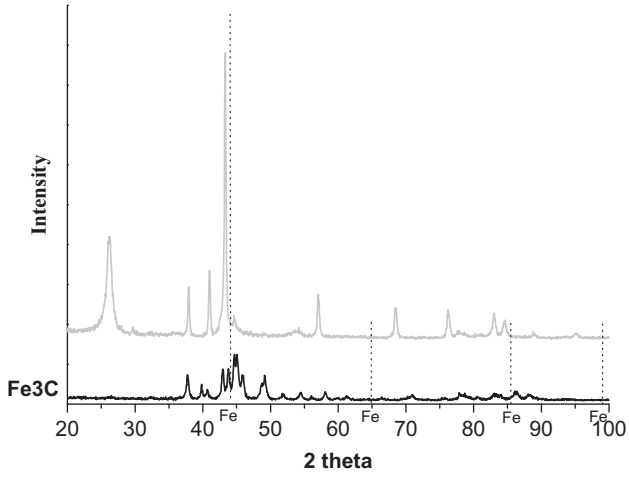

Fig. 2. X-ray diffractometer of fraction crystallite iron carbide (Fe_3C) and carbon matrix (top).

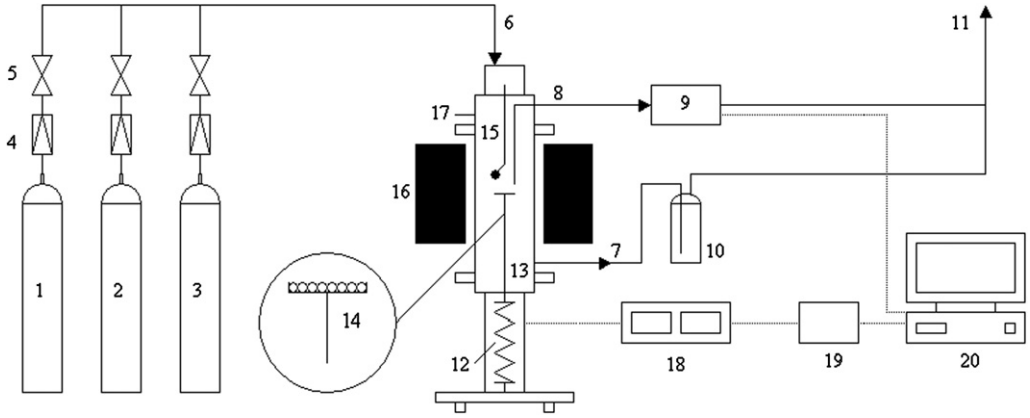

Fig. 1. Carbonizing process of nano-crystalline iron: 1—bottle with ammonia; 2—bottle with hydrogen; 3—bottle with methane; 4—pressure reducing valve; 5—flowmeter; 6—gas intake; 7—gas outlet; 8—measurement of hydrogen concentration; 9—hydrogen analyzers; 10—manostat; 11—exstractor; 12—measurement of mass change; 13—construction element; 14—platin basket; 15—thermoelement; 16—reactor oven; 17—quartz tube; 18—programator; 19—transmitter; 20—computer.