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Influence of co-solvent hydroxyl group number on properties of water-based conductive carbon pastes

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ABSTRACT

A series of water-based conductive carbon pastes were prepared by wet ball milling, followed by vacuum defoaming using isopropyl alcohol, propylene glycol or glycerin as co-solvents. Screen printing was then used to prepare conductive patterns. To determine the influence of co-solvent hydroxyl group number on the properties of water-based conductive carbon pastes, the rheological properties of the pastes and the surface morphologies and conductivities of the printed patterns were characterized. The results show that paste viscosity increased with the number of hydroxyl groups and the latter also affected thixotropy. In addition, the boiling points and surface tensions of the co-solvents increased consistently with hydroxyl group number, affecting the hydrodynamic flow. The conductive carbon paste created using propylene glycol as a co-solvent was the best for screen printing because of its weak coffee-ring effect and appropriate rheological properties, resulting in a smooth coating surface and uniform deposition of the fillers. The resistivity of the pattern printed using paste PG, containing the closest packing of conductive carbon black particles, was 0.44 Ω cm.

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Introduction

Traditional fabrication methods for conductive patterns such as electroplating and etching processes are time consuming, expensive, and complicated, which greatly limits their practical applications. Recently, screen printing has received increased attention, because it can shorten the fabrication process. Screen printing offers attractive features of direct writing and scalability to large-area manufacturing, and can be used for microelectronic device manufacturing in a variety of fields, including electronic circuits, chemical sensors, fuel cells, and organic thin film transistors (Burnside et al., 2000; Crouch, Cowell, Hoskins, Pittson, & Hart, 2005; Jewell, Hamblyn, Claypole, & Gethin, 2013; Kamyshny & Magdassi, 2014; Khaled, Mohamed, & Awad, 2008; Philip, Jewell, Greenwood, & Weirman, 2016; Secor & Hersam, 2015). Conductive pastes, which are indispensable components of the screen printing process, greatly affect screen printing quality and manufacturing costs. Compared with other kinds of conductive pastes (Bharathan

& Yang, 1998; Nur, Song, Evans, & Edirisinghe, 2002; Perelaer, de Gans, & Schubert, 2006; Smith, Shin, Stringer, Derby, & Reis, 2006), conductive carbon paste stands out as a promising material because of its low production cost and controllable electronic properties (Abhinav, Rao, Karthik, & Singh, 2015; Rao, Abhinav, Karthik, & Singh, 2015). Until now, commercially available conductive pastes have mostly been based on organic solvents, thus resulting in the emission of volatile organic compounds (VOCs). To reduce the emission of VOCs and meet increasingly stringent environmental requirements, an environmentally friendly, water-based conductive carbon paste is highly desirable (Faddoul, Reverdy-Bruas, & Blayo, 2012; Li et al., 2012; Yasin, Guo, & Demopoulos, 2016; Zhai, Zhang, Guo, Fang, & Wei, 2013).

Although water plays a crucial role in dissolving binder resins and adjusting the rheological properties of the carbon paste, the following factors restrict the wide application of water-based conductive carbon pastes. (1) Nonpolar conductive fillers, such as carbon black and graphite, tend to aggregate in the strong polar solvent, which make them difficult to disperse in the water-based system (Yasin et al., 2016). (2) Water-based conductive carbon pastes blister easily and the resulting bubbles are difficult to rupture owing to their high surface tension (Denkov, 2004;

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Vardar-Sukan, 1998), while foam is unfavorable for printing. (3) Water-based conductive carbon pastes readily block the screen mesh because of the rapid evaporation of water (Faddoul, Reverdy-Bruas, & Bourel, 2012). (4) The rheological behavior of a paste using water as the only solvent is not appropriate for screen printing. Driven by these considerations, many researchers have focused on studying the effects of various water-soluble solvents on the paste and the performance of the printed patterns (Jeong, Kim, & Moon, 2008; Kim, Jeong, Park, & Moon, 2006; Lee, Suryanarayanan, & Ho, 2009; Lin, Chang, Hwu, & Ger, 2008). However, the effects of the chemical structure or functional groups of the solvents on the properties of conductive pastes have not yet been fully investigated. The carrier of the conductive paste not only affects rheological behavior, but also the inner hydrodynamic flow of the droplet through the screen mesh. It has been reported that suspended particulate matters within an evaporating droplet prefer to deposit along the periphery and form an inhomogeneous morphology, in what is known as the “coffee-ring effect”. This effect is not conducive to obtaining uniform and high-resolution patterns (de Gans & Schubert, 2004; Deegan et al., 1997; Denkov et al., 1993; Kuang, Wang, & Song, 2014; Shen, Ho, & Wong, 2010; Still, Yunker, & Yodh, 2012; Yunker, Still, Lohr, & Yodh, 2011). The optimal strategy for eliminating the coffee-ring effect is to boost the Marangoni flow by incorporating a solvent with a high boiling point and low surface tension (Jeong et al., 2011; Lim et al., 2008).

In this study, eco-friendly alcohol co-solvents were adopted to improve the properties of conductive carbon pastes because of their excellent solubilities in water and their role in improving the dissolution of resins and the dispersion of conductive fillers. Isopropyl alcohol, propylene glycol, and glycerol were used as co-solvents, which differ in the number of hydroxyl groups present. The rheological properties and drying rates of the conductive carbon pastes were adjusted in previous work by changing the amount and type of alcohol added (Yasin et al., 2016). The present work focuses on the rheological behaviors and electrical resistivities of synthesized water-based conductive carbon pastes, and correlates them with their micro structure and morphology. The objective of this work was to correlate the number of hydroxyl groups in the co-solvents with the rheological behaviors and inner hydrodynamic flows of the conductive pastes, which greatly affect screen printing adaptability, and the surface morphology and microstructure of the printed patterns. The effect of the solvent chemical structure on the performance of the paste was also studied. This work provides insights into the selection of various solvents as carriers for the production of conductive paste.

Experimental

Materials

Conductive carbon black particles, EC-300J ($S_{\text{BET}} = 800 \text{ m}^2/\text{g}$), were purchased from AkzoNobel (Amsterdam, Netherlands). Water based acrylic resin emulsion (BS, $M_w = 70,000 \text{ g/mol}$) was provided by Shandong Baoyea Chemical Co., Ltd. (Zibo, China). Isopropyl alcohol ($\text{C}_3\text{H}_8\text{O}$, 99.7% pure), propylene glycol ($\text{C}_3\text{H}_8\text{O}_2$, 99.7% pure), and glycerol ($\text{C}_3\text{H}_8\text{O}_3$, 99.7% pure) were supplied by Guangzhou Kell Chemical Science and Technology Co., Ltd. (Guangzhou, China). Dispersive agent (sodium polyacrylate, P-19) and antifoaming agent (aliphatic mineral oil and emulsifier, SPA-202) were provided by Mandarin Shanghai Chemical Pigment Co., Ltd. (Shanghai, China). Cross linking agent (organic silicon, XR-501) was supplied by Shanghai Xirun Chemical Technology Co., Ltd. (Shanghai, China). Thickening agent (carboxyl methyl cellulose, CMC 2200) was purchased from Daicel Corporation (Osaka, Japan). Pastes and all the above chemicals were used without fur-

Table 1
Physical and chemical parameters of different solvents.

| Materials | Surface tension (mN/m) | Boiling point ($^{\circ}\text{C}$) | Viscosity (mPa s) |
|-------------------|------------------------|--------------------------------------|-------------------|
| Water | 72.8 | 100.0 | 1.0 |
| Isopropyl alcohol | 21.7 | 82.5 | 2.4 |
| Propylene glycol | 47.4 | 188.2 | 60.5 |
| Glycerol | 64.0 | 290.9 | 945.0 |

ther purification. The viscosity and surface tension of solvents are summarized in Table 1.

Preparation of the pastes

To prepare the conductive carbon pastes, first a mixture of water/co-solvent (9:1 weight ratio) was prepared. Then, dispersing, antifoaming, and cross-linking agents were added in additive quantities (0.4 wt%). The next steps were the addition of carbon black (8 wt%) that had been dried in a drum wind dryer at 85°C for 6 h, acrylic binders (3.6 wt%), and thickening agent (0.5 wt%). The remaining components of the conductive carbon paste were prepared in a mixture of water/co-solvent (9:1 weight ratio). Pastes were stirred with a glass rod and the prepared pastes were then subjected to planetary ball milling (purchased from Tencan Powder Technology Co., Ltd., Changsha, China) at a rotation speed of 300 rpm for 2 h. The volume of the zirconia jar was 500 mL and the grinding media consisted of two types of zirconia balls with diameters of 3 and 5 mm, respectively. After ball milling, the mixture was poured into a stainless steel sieve (32-mesh) to remove the grinding media. The pastes were then placed into a vacuum mixer to eliminate foam at -0.8 bar for 20 min at a rotation speed of 320 rpm. Pastes were marked as IPA, PG, and GL when using isopropyl alcohol, propylene glycol, and glycerol as co-solvents, respectively.

Screen printing tests were performed using a manual screen printer on Polyimide (PI) substrates. A polyester mesh (325-mesh with a diameter of $90 \mu\text{m}$ and thread diameter of $24 \mu\text{m}$) was used to carry out printing tests. A 17-cm-long polymer squeegee forming a 60° angle with the screen and having 70–75 shore hardness was used. A PI film ($15 \text{ cm} \times 10 \text{ cm} \times 0.0125 \text{ cm}$) was first washed by alcohol, then used as a substrate. The patterns printed on the PI substrate were a $4 \text{ cm} \times 4 \text{ cm}$ square, a circle ($\phi 4 \text{ cm}$) and three 10-cm-long lines with widths 1, 2, and 3 mm, respectively. Five min after screen printing, the PI films with printed patterns were put into the drum wind dryer at 85°C for 1 h.

Characterizations

Rheological behavior

A plate-plate rheometer (MARS60, Haake, Germany) was used to measure the rheological behavior of the pastes at shear rates varying from 0.01 to 1000 s^{-1} . The gap between the parallel plates ($\phi 35 \text{ mm}$) was equal to 1 mm and measurements were performed at 25°C . To further characterize the pastes, a thixotropic study was performed. The thixotropic tests were performed within three intervals. First, a shear rate increased from 0 to 200 s^{-1} was applied over 60 s. The shear rate was maintained at 200 s^{-1} for 10 s. Finally, the shear rate was decreased from 200 to 0 s^{-1} over 60 s. Furthermore, an oscillatory shear study was performed. The amplitude sweep study was performed at a constant frequency of 1 Hz and the shear strain was changed from 0.1% to 10% over 100 s, to determine the linear viscoelastic (LVE) region. In the LVE region, a frequency sweep study was used to characterize the rheological behavior of the paste and the frequency changed from 0.1 to 100 Hz. Prior to

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