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Preparation and Characterization of Epoxy – Silver Composite

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Abstract – Silver Ag colloids particles were chemically synthesized by modifed Tollens method. The Ag colloids aqueous solutions were characterized by UV-visible absorption spectroscopy. By using TETA (triethylenetetramine) as curing agent, Epoxy - silver composite can be made through the curing process at ambient temperature (25^0C) during 24 hours. The bulk conductivity of the Epoxy-Ag composites increased with the increase of Ag filler loadings. The morphological study of the epoxy – Ag composite with 50 phr Ag filler loadings shows of Ag particle network.

The electrically conductive adhesives (ECAs) are a new generation of material for microelectronic. The ECAs consist of a polymer binder (epoxy, silicon and polyimide) that provides mechanical strength, greater flexibility, creep resistance, energy damping and conduct electricity typically through the addition of conductive or metallic fillers, such as gold, silver, nickel, and copper. [1] Several methods have been developed to prepare Ag colloids particles. [2]. There are number of factors that affecting the final properties of ECA such as filler concentration, filler shape and size, curing time and temperature and the presence of additive or solvent in the composite system. [1]

For the synthesis of Ag colloids particles were first prepared different separate aqueous solutions, 0,46 M $AgNO_3$, 2,5 M NaOH, 0,57 M NH_4OH, 0,46 M Sucrose. The Ag colloids were prepared in a single-step chemical reduction of the complex cation $[Ag(NH_3)_2]^+$ by sucrose. The Ag colloidal particles were characterized by UV- vis absorption spectroscopy (Varian Model Cary 50 Scan UV-vis spectrophotometer). The diglycidyl ether of bisphenol-A (DGEBA; DER 331 from Dow Chemicals, Brazil), with an epoxide equivalent weigh equal to 187.5 g $eq.^{-1}$, was carefully dehydrated before use. The aliphatic amine epoxide hardeners triethylenetetramine (TETA; DEH 24 from Dow Chemical, Brazil). The Epoxy – Ag composite were prepared by mechanical stirrer of Ag particles in the epoxy resin and carefully weighing the epoxy amine hardener at the stoichiometric amount. Then the mixtures were cured during 24 h at room temperature (25 °C). Light microscope (LM) model Nikon Eclipse E200 fitted with a Nikon E 8400 digital camera analysis system had been used to study the microstructure of Epoxy – Ag composite. The electrical DC conductivity was carried out using the standard two wire on Keithley electrometer 6512.

The UV–vis absorption spectra of silver colloid particles (Figure 1) reveal the formation of silver nanoparticles by showing surface plasmon absorption maxima at 445 nm. As can be seen in the Figure 2, the bulk conductivity of the composites increased with the increase of Ag filler loadings. The curve indicates a typical conductivity curve for composites. The insulator–conductor transition was found to 20 phr of Ag filler. The higher insulator–conductor transition value and lower conductive values might be due to the composite preparation technique (using mechanical stirrer). The morphological study of the epoxy – Ag composite with 50 phr Ag filler loadings is shown in Figure 3. The Ag colloids particles dispersed in the epoxy resin are sufficient enough to have intimate contact, resulting in a Ag particle network. This network is essentially a continuous linkage between Ag particles, connecting from all direction.

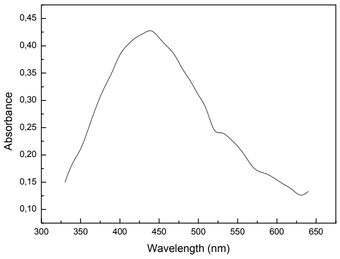

Figure 1: UV-Vis spectra of silver colloids.

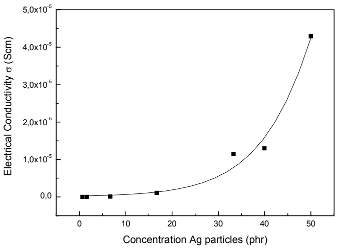

Figure 2: DC conductivity of Epoxy-Ag composite versus concentration of Ag particles (phr).

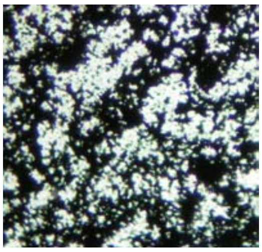

Figure 3: Optical micrographs on superficial section of Epoxy-Ag composite at filler loading of 50 phr (magnication 100x).

[1] D.I. Tee, M. Mariatti, A. Azizan, C.H. See, K.F. Chong. Composites Science and Technology 67, (2007) 2584–2591.
[2] J. Soukupova, L. Kvítek, A. Panacek, T. Nevecna, R. Zboril. Materials Chemistry and Physics 111, (2008) 77–81