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Effect of Diluent on Ultra-low Temperature Curable Conductive Silver Adhesive

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Abstract. The ultra-low temperature curable conductive silver adhesive needed urgently for the surface conductive treatment of piezoelectric composite material. The effect of diluent acetone on ultra-low temperature curable conductive silver adhesive were investigated for surface conductive treatment of piezoelectric composite material. In order to improve the operability and extend the life of the conductive adhesive, the diluent was added to dissolve and disperse conductive adhesive. With the increase of the content of diluent, the volume resistivity of conductive adhesive decreased at first and then increased, and the shear strength increased at first and then decreased. When the acetone content is 10%, the silver flaky bonded together, arranged the neatest, the smallest gap, the most closely connected, the surface can form a complete conductive network, and the volume resistivity is 2.37 × 10-4Ω · cm, the shear strength is 5.13MPa.

1. Introduction

Piezoelectric composite materials are widely used in transducers for acoustics, medical and many other fields [1-3]. The adhesion strength of the electrode layers for the piezoelectric composite transducers is a difficult research point due to the polymer components in the composite material cannot bear high temperature during the preparation of the transducer, requiring low curing temperature and short curing time conductive adhesive which also shows strong conductive ability and adhesion strength. Conductive silver adhesive had been widely used and rapid development in the field of IT and materials [4-10]. X. S. Li [10] prepared silver nanowires as conductive adhesive for the conductive filler by alcohothermal method, and it has good electrical and mechanical properties. However, currently reported conductive silver adhesive showing the curing temperature of 150 ~ 300 °C [11-17], which is a too much higher than the temperature suitable for the surface conductive treatment of piezoelectric composite material. In this paper, we prepared ultra-low temperature fast curing with high adhesion strength conductive silver adhesive, and the effect of diluent on the properties were investigated.

2. Experimental section

The resin matrix solution is firstly prepared by Epoxy Resin (AG-80), curing agent Ethylenediamine (EDA), Dibutyl Phthalate (DBP, C16H22O4, Analytical Pure), Carboxylated- terminated liquid acrylonitrile rubber (CTBN, Analytical Pure), Silane (Analytical Pure), Silane Coupling Agent (SCA-403, Analytical Pure) and accelerator Diethylene Glycol Gutyl Ether (DGBE, Analytical Pure) in proportion of 100:160:10:10:10:10. Then add acetone (Analytical Pure), prepared resin matrix solution, and nano-silver (100 nm flaky pellets) in proportion of (5~18):30:60 to obtain the final ultra-low temperature curable conductive silver adhesive. The surface morphological was observed using a Scanning Electron Microscope (SEM: EVO-18, ZEISS, Germany).

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3. Results and discussions

Figure 1 shows the diagram of the key preparing process to prepare the ultra-low curing temperature conductive silver adhesive. The ultra-low temperature curable conductive silver adhesive is prepared by two-step method. The first step is to prepare resin matrix solution and the second step is to prepare final conductive silver adhesive. The epoxy resin, curing agent, and additives were mixed in proportion of 100:150:10. And then stir the mixture was at room temperature to obtain the corresponding solution, which would then be deformed for 8 to 10 minutes. The obtained resin matrix solution would be mixed with silver powder and acetone solution. Then, stir the slurry by a constant temperature magnetic stirrer. The final conductive adhesive was obtained by vacuum treatment of stirred homogeneity mixture slurry. The prepared conductive silver adhesive was coated evenly on the surface of the piezoelectric composite material which was then putted into the vacuum box to be cured at 90 °C for 30 minutes with different dose of diluent.

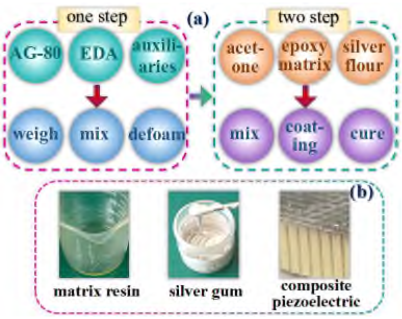
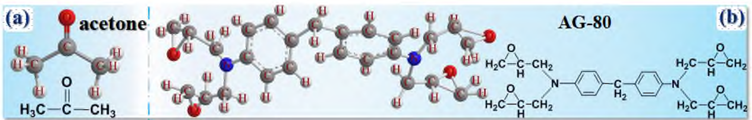


Figure 1. The diagram of the key preparing process to prepare the ultra-low curing temperature conductive silver adhesive.

Figure 2 (a) and (b) shows the molecular structure of acetone and AG-80, respectively. Figure 2 (c) shows the chemical and physical properties of acetone and AG-80, respectively. The viscosity of Ag-80 increase from 2000mPa·s to 3000~5000mPa·s when the temperature increase to 50°C. In order to improve the operability and extend the life of the conductive adhesive, the diluent was added to dissolve and disperse conductive adhesive, and reduce the viscosity of conductive adhesive, improve the coating ability and fluidity. Acetone is an inactive diluent, does not participate in the curing reaction, and will evaporate during the curing process, so that increasing the shrinkage of resin.



(c)

properties	Average molecular weight	Viscosity (25℃)/mPa·s	Viscosity (50℃) /mPa·s	volatile (%)	epoxy value (mol)	tensile modulus (GPa)
AG-80	422	2000	3000～5000	<1.5	0.8	3.7
properties	Average molecular weight	Viscosity (20℃)/mPa·s	volatilization rate (nBAc=1.25℃)	Solubility parameter $\delta/(cal\cdot cm^{-3})^{1/2}$	flash point (℃)	surface tension (dyne/cm)
acetone	58.08	0.316	5.7	20.3	-20	18.8

Figure 2. The chemical structural formula, characteristics contrast of AG-80 and 618. (a) and (b) shows the molecular structure of acetone and AG-80, respectively; (c) shows the chemical and physical properties of acetone and AG-80, respectively.

Figure 3 shows the volume resistivity and shear strength of conductive adhesive with different content of the diluent. It can be seen from the figure that with the increase of the content of diluent, the volume resistivity of conductive adhesive decreased at first and then increased, and the shear strength increased at first and then decreased. When the acetone content is 10%, the volume resistivity is $2.37 \times 10^{-4}\Omega \cdot cm$ and the shear strength is 5.13MPa. When the acetone content is 8%, the volume resistivity is $8.06 \times 10^{-4}\Omega \cdot cm$ and the shear strength is 6.54MPa. This is due to the addition of a small amount of diluent can improve the shrinkage of the resin, making the contact between the silver powder is better, improving the conductivity of the conductive adhesive, and can increase the cohesive energy density of the resin system and enhance the mechanical properties of the resin system. However, the added too much diluent will affect the curing reaction, resulting in the decrease of contact of conductive adhesive particles, reduce the conductivity. At the same time, the resin is over crosslinked, resulting the resin system becomes brittle, the rigidity is weakened and the shear strength is reduced.

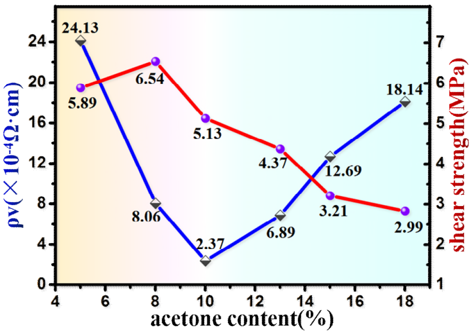


Figure 3. The volume resistivity and shear strength of conductive adhesive with different content of the diluent.

Figure 4 shows the SEM images of conductive silver adhesive with different content of diluent. It can be seen that when the diluent content of 10%, the silver flaky bonded together, arranged the neatest, the smallest gap, the most closely connected, and the surface can form a complete conductive network. This is due to adding diluent can reduce the resin viscosity, enhance the resin shrinkage, so that closer contact between the silver. When the amount of diluent is too small, the matrix resin is too thick, resulting in silver powder is not open, and forming a large number of porous microstructure. Furthermore, the addition of diluent affects the curing reaction to a certain extent. When the amount of diluent is too much, the conductive silver adhesive cannot be a good curing, affecting the uniform distribution of silver powder, reduce the conductivity. This also explains why the conductive resist has the smallest volume resistivity at a diluent content of 10%.

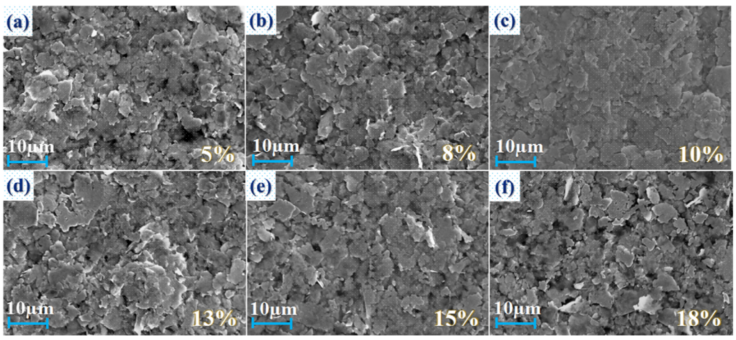


Figure 4. The SEM images of conductive silver adhesive with different content of diluent.

4. Conclusions

In summary, the effect of diluent acetone on ultra-low temperature curable conductive silver adhesive were investigated for surface conductive treatment of piezoelectric composite material. In order to improve the operability and extend the life of the conductive adhesive, the diluent was added to dissolve and disperse conductive adhesive. With the increase of the content of diluent, the volume resistivity of conductive adhesive decreased at first and then increased, and the shear strength increased at first and then decreased. When the acetone content is 10%, the silver flaky bonded together, arranged the neatest, the smallest gap, the most closely connected, the surface can form a complete conductive network, and the volume resistivity is $2.37 \times 10^{-4}\Omega \cdot$ cm, the shear strength is 5.13MPa.

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