Research on the related properties of ACF anisotropic conductive adhesive

Pengyi Qiao [a], Maoniu Liu [b], Rui Wu [c], Haoxun Yao [d], Xiaoshuai Xing [e], Yingxia Liu [f], Xiuchen Zhao [g]

Beijing Institute of Technology, Beijing,China

[a] Feanaro_Curufinwe@163.com, [b] bitlmn6966@163.com, [c] wurui@semi.ac.cn, [d] rainyhx@foxmail.com, [e] 15503625348@163.com, [f] yingxia.liu@bit.edu.cn, [g] zhaoxiuchen@bit.edu.cn

Abstract. Bonding strength is an important indicator to measure the life of the package in the process of Electronic Packaging. Anisotropic conductive adhesive film is a kind of package bonding. In this paper, scanning electron microscopy is used to compare the microstructure and element distribution of two different types of conductive adhesives CP6920F and HA5125PL both before and after flip-chip curing. At the same time, the 280SJ four-point probe sheet resistance tester and Bruker Icon atomic force microscope are used to measure the electrical properties of the cured conductive adhesive, and we use INSTRON5985 electronic universal material testing machine to test the shear strength and tensile strength of the cured conductive adhesive film.

Keywords: Anisotropic Conductive Adhesive, heat curing, Microstructure, performance study.

1. Introduction

With the rapid development of electronic products towards lighter, thinner, shorter and smaller size, the use of anisotropic conductive adhesive (ACA) has also gained more attention to complete the inter-connection between the substrate and the flip chip, and it has been applied in high-density IC packaging such as liquid crystal display drivers, handheld computer display driving circuits, mobile electronic products and board chips [1]. Anisotropic Conductive Adhesive Film (ACF), is film-like, which belongs to a kind of ACA, because of its continuous processing and the characteristics of extremely low material loss, it is increasingly and widely used in electronic packaging [2-5].

Anisotropic conductive adhesives are generally divided into thermosetting and thermoplastic anisotropic conductive adhesives. Most of the thermosetting anisotropic conductive adhesives currently used are thermosetting anisotropic conductive adhesives due to the higher strength than that of thermoplastic anisotropic conductive adhesives. Thermose-tting anisotropic conductive adhesive is a film formed by mixing a certain amount (generally about 3% to 15% by volume) of conductive particles in an organic polymer matrix (such as epoxy resin). The surface of the conductive particles generally has a layer of Ni/Au coating, and some conductive particles also have an insulating film on the outside, with a size of about 2 to 10 microns. Since the conductive particles are generally spherical and the outer layers are blocked by insulating resins, the ACF itself does not conduct electricity. During the bonding process, due to the presence of certain pressure and temperature, the ACF will show a softened state, as well as the conductive particles flow slowly and are randomly and uniformly distributed. The insulating film of the outer layer of the conductive particles subjected to the pressure is ruptured, and at the same time, the electrical connection in the direction parallel to the direction of the pressure is realized. In other directions, it is still non-conductive because of the existed insulating film. As a result, the unidirectional conduction in the Z axis direction and the insulating bonding effect in the X and Y axis directions are achieved, respectively.

At present, the common ACF brands include Sony, Hitachi Chemical, Weifeng, Haizheng Industrial, etc. The products of each brand are generally similar in functionality, but there are still differences. The main products used in this paper are the products processed by Hitachi Chemical and domestic Haizheng Industrial for analysis and comparison. The current mainstream research

mainly focuses on the improvement of its basic properties (such as bond strength) and special properties (such as low hygroscopicity, low stress), the composition and structure of the conductive particles, the composition of the binder, and the effects of the curing process on the properties of the anisotropic conductive adhesive film were studied.

In view of the current development of ACF, it is necessary to start a new system. Most of the conductive adhesive films currently used are epoxy resin systems. Epoxy resins have disadvantages such as high curing temperature and strong hygroscopicity. Compared with the Pb/Sn system, the bonding strength is lower than that of the Pb/Sn system, which cannot fully meet the requirements of the electronics industry. The development of the new system will surely make it more widely used in the electronics industry [7]. In addition, many scholars have carried out research work to develop new structural ACFs to improve the reliability of ACF connectors. Scholars such as Eom prepared a new type of anisotropic conductive adhesive film composed of a polymer as a matrix and a low melting point solder doping combination. Yim et al. [8] developed a new type of anisotropic conductive adhesive film with a multilayer structure. Compared with the traditional ACF, the conductive adhesive added two layers of insulating functional resin layers above and below the colloid. Improve the bonding performance of conductive adhesive with chips and substrates and control the flow of conductive particles during curing.

This paper mainly studies the performance of two different types of conductive adhesives CP6920F and HA5125PL after the flip-chip thermosetting process in the form of stepped heating. Firstly, the microstructure and element distribution of the two conductive adhesives CP6920F and HA5125PL before curing were analyzed by scanning electron microscope, then use flip-chip hot-pressing equipment to flip-chip bond copper block samples with ACF conductive adhesive under two stages of preheating and main hot-pressing, the microstructure and element distribution of the cured conductive adhesive were analyzed and compared by scanning electron microscopy. At the same time, the 280SJ four-point probe sheet resistance tester and Bruker Icon atomic force microscope were used to measure the electrical properties of the cured conductive adhesive, and use INSTRON5985 electronic universal material testing machine to test the shear strength and tensile strength of the cured conductive adhesive film.

The remaining paper is organized as follows. Section II introduces the experimental program and the experimental results are shown in Section III.

2. Experimental program

2.1 Hot press curing

The main hot-pressing curing equipment used in this experiment is a Flip Chip hot-pressing equipment. The hot-pressing equipment is set to the preheating temperature and main heat temperature of the corresponding conductive adhesive in the form of stepped heating, and the pressurization system of the equipment is adjusted to apply corresponding pressure to the conductive adhesive during the heating process. The bonding process parameters of the two conductive adhesives are shown in Table 1 and Table 2.

Table 1. HA5125PL hot pressing parameters

hot pressing stage	*Table column subhead*	*Subhead*	*Subhead*
preheat pressure	50~80℃	1~2s	0.5~1.0MPa
main heat press	180±10℃	15±5s	2.0~4.0MPa

Table 2. CP6920F hot pressing parameters

hot pressing stage	*Table column subhead*	*Subhead*	*Subhead*
preheat pressure	60~80℃	1~2s	0.3~1.0MPa
main heat press	190℃	20s	0.2~0.4MPa

2.2 Test and Characterization Means

1) Scanning electron microscope

According to the above hot pressing conditions, using flip-chip hot pressing equipment, the conductive adhesive is cured by hot pressing with the copper sheet as the base, and the white separator is removed from the conductive adhesive after hot pressing, so that the cured conductive adhesive layer exposed.

Scanning Electron Microscope (SEM) uses an electron beam to focus on the surface of the sample. The high-energy, finely focused electron beam scans on the surface of the sample under the action of a scanning coil, interacts with the sample, and generates various physical signals. The intensity of various signals is related to the surface features (topography, composition, structure, etc.) of the sample, which can be detected, amplified, and imaged by different detectors, and used for various microscopic analysis. The main signal collected by scanning electron microscopy are secondary electrons and backscattered electrons. Since the conductivity of the conductive adhesive is anisotropic, that is, the X-Y plane is in an insulating state, in order to increase the conductivity of the sample and facilitate SEM observation, the surface is treated with carbon spray.

In this experiment, a cold field emission high-resolution scanning electron microscope (HITACHI SU8220) was used. The cold field emission high-resolution scanning electron microscope is equipped with a new field emission electron gun, which makes the electron beam current larger and more stable, and has ultra-high resolution capability. The secondary electron resolution at low acceleration voltage (1kV) can reach 1.1nm. Equipped with a Bruker FlatQuad type flat-insertion X-ray spectrometer, it can perform high-resolution elemental analysis on samples. Applied to the structure and composition analysis of nanoscale materials such as biology and chemistry.

2) Four probe sheet resistance test

The purpose of this experiment is to verify the anisotropic conductive properties of the conductive adhesive, that is, to verify that the X-Y plane of the conductive adhesive after thermosetting is in an insulating state. Four probes were pressed vertically on the X-Y surface of the cured conductive adhesive, and a DC current I was passed through the sample between the two outer probes, and the potential difference V generated between the inner two probes was measured [8]. According to the displayed value, it can be judged that the X-Y plane of the conductive adhesive is in an insulating state. In this experiment, a 280SJ four-probe sheet resistance tester was used.

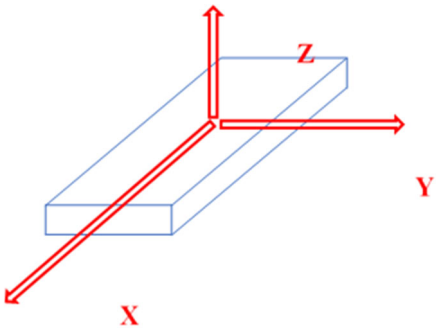


Fig. 1 3D schematic diagram of conductive adhesive

3) Scanning Probe Microscope System

In order to measure the conductivity of the conductive adhesive in the Z-axis direction, scanning probe microscopic measurement was carried out in this direction. The scanning probe microscope system consists of an atomic force microscope, an operating computer and an electronic control system. After curing the conductive adhesive on the silicon wafer, place it on the sample stage of the atomic force microscope, and make the probe vertically contact the surface of the conductive adhesive.

AFM can accurately observe the three-dimensional morphology of sample surface micro-area (nano- and micro-scale) in atmospheric environment; at the same time, it can study the physical

properties (electrical, magnetic, mechanical, etc.) of the sample surface, and can test a variety of materials such as Insulating materials, semiconductor materials, membrane materials, piezoelectric/ferroelectric materials, low-dimensional nanomaterials, etc.

The Dimension Icon scanning probe microscope system was used in this experiment, and the atomic force microscope was Bruker Icon. The XYZ axis scanning range is 90μm*90μm*10μm, the XY direction resolution is about 1nm (related to probe selection), and the Z direction resolution is 0.1nm.

4) Electronic universal testing machine and muffle furnace

In order to measure the shear and tensile strength of the conductive adhesive after curing, after cleaning the adhesive surface of the brass sample, first apply the conductive adhesive to the pre-set adhesive surface, as shown in Fig. 2. Then use flip-chip welding equipment to heat and press the sheared sample to solidify, as shown in Fig. 3. Because the volume of the tensile sample is too large, in this experiment, flip-chip welding equipment was used to preheat and press it. Put into a muffle furnace for heat gluing. Finally, put the cured shear and tensile samples into the universal material testing machine for measurement.

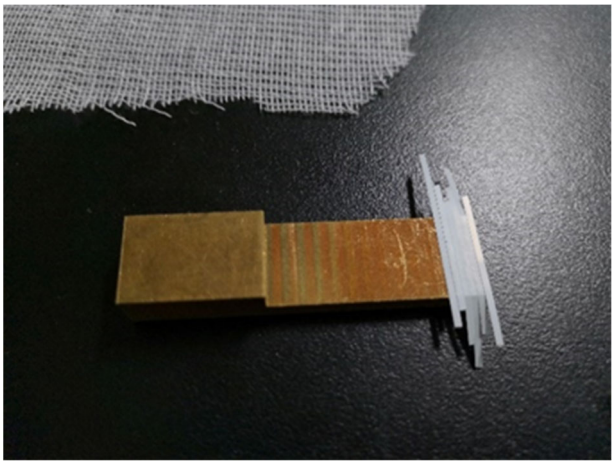


Fig. 2 Glue the sample.

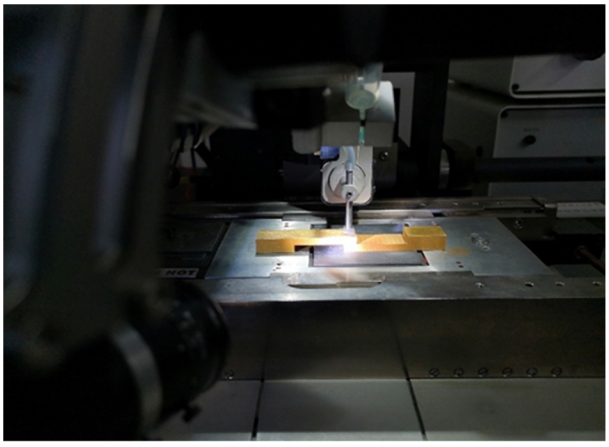


Fig. 3 Heat press the shear specimen.

3. Experimental Result

3.1 Microscopic element distribution of two conductive adhesive films

In this paper, two types of conductive adhesives are studied. The first is the study of surface morphology and element content. According to the experimental results of scanning electron microscope, the matrix of CP6920F conductive adhesive is an organic resin material containing Si,

and the conductive particles are nickel-based ball gold-plated layers. The matrix material of HA5125PL conductive adhesive is also an organic resin material containing Si, and the conductive particles are Nickel and gold layers plated sequentially on the surface of organic spheres. Fig. 4 and Fig. 5 show the Z direction and the element content distribution in this direction of the two conductive adhesive films before curing.

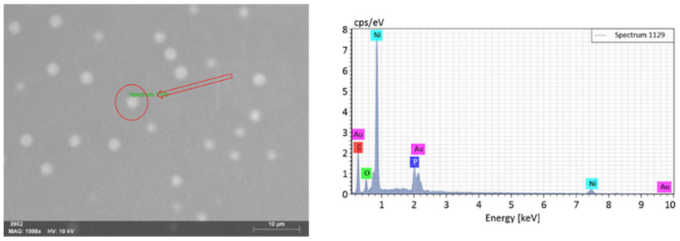


Fig. 4 Z direction of CP6920F conductive adhesive film and distribution of element content in this direction

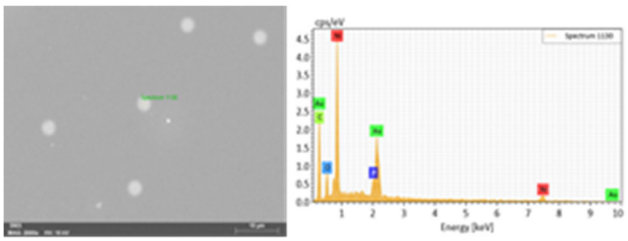
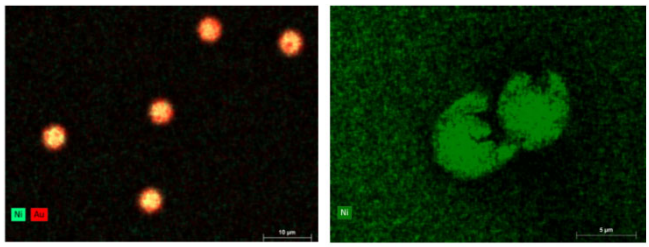


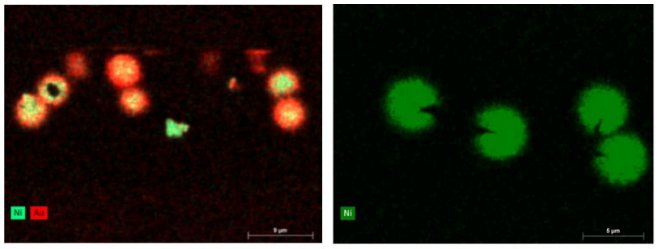
Fig. 5 Z direction of HA5125PL conductive adhesive film and distribution of element content in this direction.

The comparison pictures of the two conductive adhesives before and after curing are shown in Fig. 6 and Fig. 7. It can be seen from the comparison in the figure that the content of Ni in the organic carrier in the HA5125PL conductive adhesive is more than that in the CP6920F conductive adhesive. At the same time, in the electron microscope photo of CP6920F conductive adhesive before curing, Ni has fallen off, and there are voids in the middle of the metal particles in the circle. According to these analysis, the conductive adhesive particles of CP6920F are a layer of Ni and Au plated on the organic balls, while the conductive particles of HA5125PL conductive adhesive are Au plated on Ni-based balls, so after curing, due to the formation of complexes So the diffused Ni is more than that of CP6920F.



(a) Before curing (b) After curing

Fig. 6 Comparison of HA5125PL conductive adhesive before and after curing.



(a) Before curing (b) After curing

Fig. 7 Comparison of CP6920F conductive adhesive before and after curing.

According to the analysis of the experimental results of the above scanning electron microscope, the elemental compositions of the two conductive adhesives are shown in Table III and Table IV. The table on the right shows that the Au content of HA5125PL is much higher than that of CP6920F, while the Ni content is just the opposite.

Table 3. Scanning Electron Microscopy Results of CP6920F Condyctive Adhesive

element	*atomic number*	*net worth*	*quality [%]*	*Normalized mass [%]*	*atom [%]*	*error [%]*
C	6	3175	11.75	21.07	55.00	2.03
O	8	1007	2.29	4.11	8.05	0.54
P	15	2879	5.08	9.12	9.23	0.25
Ni	28	16956	25.68	46.07	24.61	3.38
Au	79	3138	10.95	19.64	3.13	0.51
Total			55.75	100	100	

Table 4. Scanning Electron Microscopy Results of HA5125PL Condyctive Adhesive

element	*atomic number*	*net worth*	*quality [%]*	*Normalized mass [%]*	*atom [%]*	*error [%]*
C	6	3325	12.07	20.38	59.91	2.07
O	8	1139	3.00	5.06	11.18	0.68
P	15	1102	2.24	3.79	4.32	0.15
Ni	28	9574	16.69	28.20	16.96	2.38
Au	79	6240	25.20	42.57	7.63	1.06
Total			59.20	100	100	

3.2 Electrical performance test

In this experiment, the sample was tested twice. The first time was to test the XY plane of the conductive adhesive by using four probes, and the second time was to use the atomic force microscope to test. At the same time, in the subsequent curing, the curing pressure parameter was increased, use a multimeter to measure the conductivity in the Z-axis direction, and the multimeter shows the number. From this phenomenon analysis, it can be concluded that the two conductive adhesives studied in this paper are indeed anisotropic conductive adhesives. They do not have conductivity in the x and y directions, but only in the z direction. In the case of pressing, when the insulating layer of the conductive particles in the conductive adhesive is not damaged, it is also unable to conduct electricity in the z direction. Sufficient pressure must be applied so that the conductive particle metal layers are in contact with each other to conduct electricity in the z-direction.

3.3 Shear force measurement test

The shear results of the two conductive adhesives measured under their respective optimal process conditions are shown in Table V.

However, in the actual test process, some areas were not fully bonded. The data acquisition of the experimental results was corrected, and the bonding area and the maximum load at failure were directly read, and the shear strength was further calculated. The corrected experiment the data are shown in Table VI.

Table 5. Shear strength results of two conductive adhesive films (Rp/MPa)

Conductive adhesive type	*first group*	*Second Group*	*The third group*	*average value*
CP6920F	2.1	2.2	5.5	3.3
HA5125PL	5.2	2.4	3.6	3.7

Table 6. The revised shear strength results of the two conductive adhesive films

Conductive adhesive type	*Load [/N]*	*Area [/mm²]*	*Shear strength [/Rp/MPa]*
CP6920F	460	39.3	11.7
HA5125PL	785	52.9	14.8

The bonding surface of the shear specimen used in the first test was relatively rough, and the second test was performed again after the surface of the shear specimen was polished and smoothed. From the results, the smooth surface makes the overall strength greater than that of the rough surface. This shows that when the bonding surface is relatively smooth, the shear strength is greater because the bonding contact area is larger and there is no surface roughness to reduce the actual bonding area. At the same time, the irregularity of the strength is considered to be closely related to the pressure during curing. Since the pressure cannot be applied accurately together, the pressure is different, resulting in different strengths.

3.4 Tensile force measurement test

The prepared samples were bonded according to their respective optimal experimental conditions, and the tensile strength was tested on a universal testing machine. The results are as follows:

Table 7. Shear strength results of two conductive adhesive films (/Rp/MPa)

Conductive adhesive type	*first group*	*Second Group*	*average value*
CP6920F	34.1	73	53.55
HA5125PL	40.3	51.2	45.75

For the tensile test, there is also no regularity in the data, especially for the domestic conductive adhesive, the data is quite different. Analyzing the reasons, it is believed that due to the influence of pressure, the tensile pattern is cured in the muffle furnace, and the applied pressure is provided by the screws at both ends of the fixture, and the pressure is not strictly controlled when tightening the screws, so the final test results are very different.

References

[1] Liu Johan. Conductive adhesives for eletronics packaging. Electrochemical Publications LT D, Port Erin, Isle of M an, British Isles, 1999

[2] Tmmala R.R, Garrou P., Gupta T.K., Microelectronics Packaging Handbook: Semiconductor Packaging, Part II. In: Tummala R.R., Rymaszewksi E.J., and Klopfenstein A.G., Ede. New York: Chapman&Hall, 1997:285-293.

[3] Yamaguchi M., Asai F., Eriguchi F., Development of novel anisotropic conductive film (ACF), The 8th IEEE Electronic Components and Technology Cnference, 1998, 360-364.

[4] Rasul J., Olson W., Flip chip on paper assembly utilizing anisotropic conductive adhesive, Proceedings of 53nd Electronic Components and Technology Conference, 2002, 90-94.

[5] Delaney D., White J., Flip chip assembly utilizing anisotropic conductive films: a feasibility study, IEEE Electronic Components and Technology Conference, 2000, 641-645.

[6] Jian Ma, Hong Gao, Xu Chen. Preparation of composite conductive microspheres used in anisotropic conductive adhesive films, Advanced Materials Research, 2011, 194-196.

[7] Liong S, Wong C P. An Alternative to Epoxy Resin for Application in Isotropically Adhesive. 2001 International Symposium on Advanced Packaging Materials. Braselton GA, 2001:13-18.

[8] Eom Y.S., Baek J.W., Moon J.T., et al., Characterization of polymer matrix and low melting point solder for anisotropi conductive film, Microelectronic Engineering, 2008,85(2):327-331.