Z-Axis Anisotropic Electrical Conductor Films in Adhesive and Standalone Forms for Electrical Interconnection

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Z-axis electrical conductor films in adhesive and standalone forms were made from nickel particles (one per conduction path) and a polymer matrix (polyvinylidene fluoride for the standalone film and epoxy for the adhesive film). The standalone film was found to exhibit resilience, conducting path pitch 125 μm and z-axis resistance 2 Ω.4 Ω, 17, 48 Ω, and 160 Ω per connection at z-axis pressures of 1.39 MPa, 0.037 MPa, 0.0046 MPa, and 0 MPa respectively; each connection consisted of one nickel particle, which protruded from both sides of the film; the film exhibited no stress relaxation due to the absence of polymer deformation under z-axis pressure. The adhesive film exhibited pitch 64 μm and z-axis resistance 52 Ω per connection at zero pressure.

Key words: Interconnection, anisotropic, electrical conductor, film, nickel, polymer, composite

INTRODUCTION

A z-axis anisotropic electrical conductor film is a film which is electrically conducting in the direction perpendicular to the film, but is insulating in all other directions. This film is technologically valuable for use as an interconnection material in electronic packaging (chip-to-package, package-to-board and board-to-board), as it electrically connects the electrical contact pads touching one side of the film with the corresponding contact pads touching the direct opposite side of the film. Even though the film is in one piece, it contains numerous z-axis conducting paths (not necessarily in a regular array, can be randomly distributed), so that it can provide numerous interconnections. If each contact pad is large enough to span a few z-axis conducting paths, no alignment is needed between the contact pad array and the z-axis film, whether the conducting paths are ordered or random in their distribution.[1–17] Under this situation, in order to attain a high density of interconnections, the cross section of each z-axis conducting path must be small. However, if each contact pad is only large enough to span one z-axis conducting path, alignment is needed between the contact pad array and the z-axis film, and this means that the conducting paths in the z-axis film must be ordered in the same way as the contact pad array.[18] An example of an application of a z-axis conductor film is in the interconnections between the leads from (or contact pads on) a surface mount electronic device and the contact pads on the substrate beneath the device. In this application, one piece of z-axis film can replace a whole array of solder joints, so processing cost can be much reduced. Furthermore, the problem of thermal fatigue of the solder joints can be avoided by this replacement. Another example is in the vertical interconnections in three-dimensional electronic packaging.

A z-axis film is a polymer-matrix composite containing conducting units which form the z-axis conducting paths. The conducting units are usually particles, such as metal particles and metal coated polymer particles. The particles can be clustered so that each cluster corresponds to one conducting path.[1–5] They can also be separate from one another, so that one particle corresponds to one conducting path.[6] In the former case, the particle size can be even less than the thickness of the film, as particles can stack up to form a z-axis conducting path.[7,8] In the latter case, the particles must be at least as large as the film thickness. Instead of being particles, the conducting units can be wires parallel to the z-axis,[7,8] but film fabrication involving wires is more difficult than that involving particles. Moreover, the conducting units can be metal columns obtained by metalization[9] or dots obtained by screen printing silver filled epoxy.[18]

The z-axis conductor film can be an adhesive or a standalone (or free-standing) film. The adhesive

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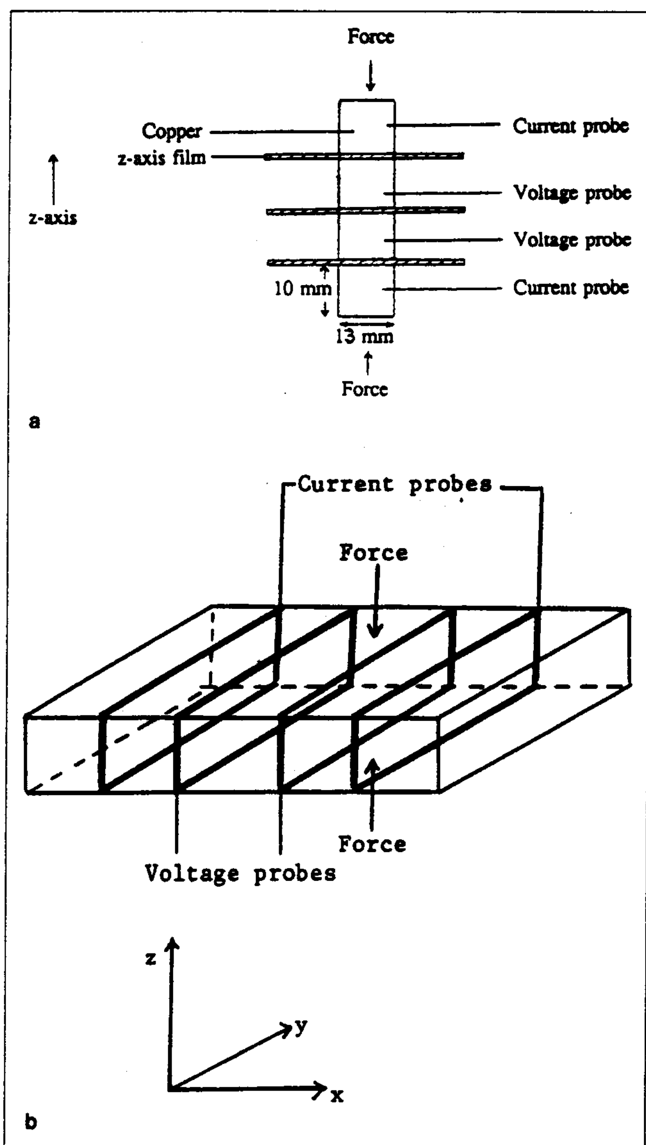

Fig. 1. Schematic illustration of method for measuring the electrical resistivity. (a) z-axis; (b) x-axis.

form[10–14] facilitates joining, but the joint cannot be easily disconnected. The standalone film requires an applied pressure, as provided by mechanical fastening, so it is less convenient for joining. However, it provides separable interconnections (i.e., interconnections which can be easily disconnected and connected back).[2,3,7,8] Since the separable film must be reusable, it must be resilient.

Previous work on separable z-axis films[2,3,6–8] used either an elastomer (such as silicone) or a thermoplast (such as polyimidesiloxane and seflon) as the polymer matrix in order to provide resilience. Metal wires (e.g., brass[7,8]), metal columns,[9] metal particle columns (e.g., gold plated nickel[7,8]) and individual metal coated polymer particles[6] had been used to provide z-axis conducting paths. Particle columns were formed by magnetic alignment of the particles. Using particle columns, Ref. 2 attained a conducting path width of 400 μm and a pitch (center-to-center distance between adjacent conducting paths) of 290 μm. Also using particle columns, Ref. 7 and 8 attained a conducting path width of ~10 μm and a pitch of ~100 μm. In general, a large conducting path width is desirable for decreasing the resistance per path, while a small pitch is desirable for high density interconnection. In contrast to the use of metal wires, mete columns or metal particle columns, this paper uses one metal particle per conducting path (i.e., per connection). The concept of one particle per path had been demonstrated in Ref. 6 by using metal coated polymer particles. However, due to the high resistivity of the metal coating compared to the bulk metal, the z-axis resistivity of the film was high (0.5 Ω · cm for a conducting path).[6] By using metal particles in place of metal coated polymer particles, this work has decreased the z-axis resistivity of a conducting path to 10^{-6} Ω·cm. Furthermore, in contrast to previous work, this work does not rely on a polymer (whether the matrix or the particles) for providing resilience, as the resilience is provided by the metal particles, which protrude from both sides of the standalone film. As a result, the problem of stress relaxation of the polymer is eliminated. In addition, the protrusion of the metal particles eliminates the problem of open circuiting the connection upon heating due to the higher thermal expansion of the polymer compared to the conductor.[15]

Most previous work on z-axis adhesive films[13,14] used an adhesive with randomly dispersed conductive particles (8–12 μm diameter) suspended in it. The particles were phenolic spheres that had been coated with nickel. After bonding under heat (180–190°C) and pressure (1.9 MPa), a particle became oval in shape (4 μm thick). There was one particle per conducting path. The main drawback of this technology is the requirement of heat and pressure for curing the adhesive. Heat and pressure are not desirable in practical use of the z-axis adhesive. In this paper, the need for heat and pressure is removed through the choice of the polymer.

A different kind of z-axis adhesive film[18] used screening or stenciling to obtain a regular two-dimensional array of silver filled epoxy conductive dots, but this technology suffers from the large pitch (1500 μm) of the dots and the consequent need for alignment between z-axis film and contact pad array. In this paper, the pitch of the conducting paths in the z-axis adhesive film is as low as 64 μm.

EXPERIMENTAL METHODS

Z-Axis Standalone Film Preparation

The z-axis films in the standalone form were fabricated by (i) mixing polymer powder (polyvinylidene fluoride or PVDF, 3-5 μm size, Kynar 721, from Elf Atochem America Inc., Philadelphia, PA) and nickel powder (62–74 μm size, Novamet Spherical Nickel Powder, Type 4SP, from Novamet Corporation, Wyckoff, NJ), (ii) hot pressing the mixture between copper foils (25 μm thick) at 180–190°C and 6.0–6.5

Table I. Electrical Resistivity in the Z-Axis of Z-Axis Conductor Standalone Films

Wt.% Ni	60		67		71			
Vol.% Ni*	23		29		33			
Area % of Ni at mid-plane of film	23		27		33			
Conducting path density (cm^{-2})	6.0×10^3		7.0×10^3		8.5×10^3			
Pitch+ (μm)	150		137		125			
Z-axis pressure (MPa)	0.037	1.39	0.037	1.39	0	0.046	0.037	1.39
Z-axis resistance within one path (Ω)	46	5.1	38	3.1	160	48	17	2.4
Z-axis resistivity[1] (Ω · cm)	6.3×10^{-2}	7.0×10^{-3}	4.4×10^{-2}	3.6×10^{-3}	$\sim 1.6 \times 10^{-1}$	4.6×10^{-2}	1.6×10^{-2}	$2.3 \times ^{-3}$
Effective conducting path diameter** (μm)	0.37	1.1	0.40	1.4	0.20	0.36	0.61	1.6

*Calculated from wt.% Ni using densities of Ni and PVDF.
+Average distance between centers of adjacent paths.
[1]For overall film.
**Calculated from the z-axis resistance within one path by assuming that each path is a nickel cylinder.

MPa for 30 min, and (iii) cooling to room temperature under pressure. Films of three compositions were studied, namely 60, 67, and 71 wt.% Ni (corresponding to 23, 29. and 33 vol.% Ni), as obtained by controlling the proportions of Ni particles and PVDF powder in the mixture. The ductility of the copper foils allowed the nickel particles to protrude slightly from both sides of the resulting film. The protrusion served to facilitate contact to be made between a z-axis conducting path and the contact pad touching a side of the film. Films fabricated without the copper foils (i.e., just with steel plates sandwiching the powder mixture) were inferior due to the smaller degree of protrusion. Chemical etching of the polymer using dimethyl sulfoxide (Sigma Chemical Co., St. Louis, MO) at 60–70°C after fabrication of the film without copper foils was found to increase the degree of protrusion, but it caused the formation of a ditch around each nickel particle. All the standalone films reported here were fabricated with copper foils and without etching.

Z-Axis Adhesive Preparation

The z-axis adhesive was fabricated by mixing polymer and nickel powder (25–32 μm size, sieved, Novamet Spherical Nickel Powder, Type 4SP, from Novamet Corporation, Wyckoff, NJ). The polymer was epoxy (Resin 813) and EPI-Cure (R) 3234 in 100:13 weight ratio, from Shell Chemical Co. (Houston, TX), together with 1 wt.% isobutanol (to lower the surface tension of the epoxy before curing and diminish the brittleness after curing). The nickel particles had been treated by ball milling in acetone for 2 h (in order to make them more regular or spherical in shape), and by immersion in dilute hydrochloric acid for 3 min (in order to remove the oxide from the nickel particle surface; such cleaning is particularly important when the particles are small) and then water rinsed and dried. The z-axis adhesive was applied to a substrate in order to form a z-axis film. No heating was needed to cure the epoxy. No pressure was applied during bonding, other than the small pressure (0.16 MPa) provided by a paper clip. In contrast to the standalone film, the nickel particles did not protrude from the surface of the film on either side. As a result of this and the size distribution of the nickel particles, the fraction of nickel particles that serve as z-axis conduction paths (with one nickel particle per path) is believed to be smaller for the adhesive film than the standalone film. Adhesives of three compositions were studied, namely 60, 67, and 71 wt.% Ni (corresponding to 17, 22, and 24 vol.% Ni), as obtained by controlling the proportions of Ni particles and epoxy resin in the mixture.

Testing

The electrical resistivity of a z-axis film was measured by using the four-probe method. For measuring the resistivity in the x-y plane, silver paint was used for the electrical contacts. For measuring the resistivity in the z-axis, silver paint was not used and three films were stacked with copper cylinders (0.5 in or 13 mm diameter, 10 mm height) in between (Fig. 1a), such that the four copper cylinders were lined up with the test area on each of the three z-axis films. The two current probes were applied to the outer pair of copper cylinders; the two voltage probes were applied to the inner pair of copper cylinders. For measuring the resistivity in the x-direction, silver paint was applied along the perimeters of four parallel y-z planes (Fig. 1b). The two current probes were applied to the outer pair of y-z planes; the two voltage probes were applied to the inner pair of y-z planes. Because the z-axis electrical resistivity of the standalone film was pressure dependent, the measurement was carried out under different constant z-axis pressures and during

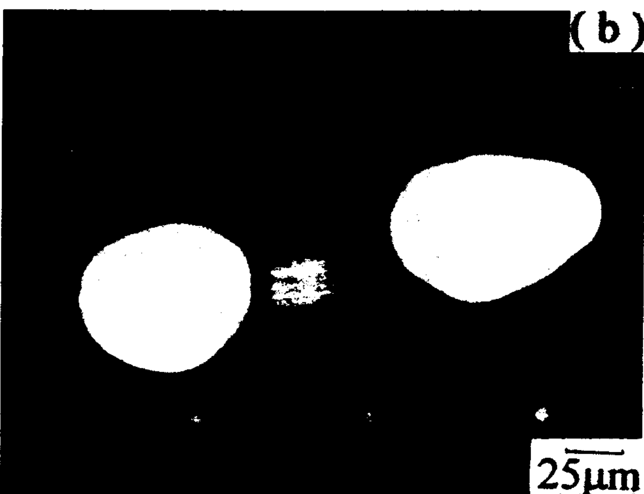

Fig. 2. Micrographs of z-axis conductor film. (a) Top view (by SEM, sample not polished); (b) Side view (by optical microscopy, sample mounted in polyester and mechanically polished).

pressure cycling. The electrical resistivity in both directions, namely the z-axis direction and the in-plane direction, were separately measured. For the z-axis adhesive film, no pressure was applied.

To investigate the stress relaxation effect, if any, the z-axis resistivity was measured as a function of time up to 2000 s at various fixed pressures up to 1.4 MPa, as provided by using a hydraulic mechanical testing system (MTS Model 810).

RESULTS

Z-Axis Standalone Film

The resistivity in the z-axis of the standalone films is shown in Table I at different constant z-axis pressures. At a given pressure, the z-axis resistivity decreased with increasing Ni content. At a given Ni content, the resistivity decreased with increasing z-axis pressure. The film with the highest Ni content (71 wt.%) gave the lowest resistivity, so it is most attractive technologically. Even at zero z-axis pressure, the film conducted along the z-axis. Table I shows the measured zaxis resistance within a conducting path (i.e., z-axis connection resistance), z-axis resistivity of the overall film, effective path diameter and pitch. The effective path diameter, obtained by assuming that each path was a nickel cylinder of resistivity $6.9 \times 10^{-6}\ \Omega \cdot$ cm, increased with increasing pressure and was much smaller than the nickel particle diameter. The resistivity in the x-y plane was 1.2×10^{9} $\Omega \cdot$ cm at zero pressure for the film with 71 wt.% Ni.

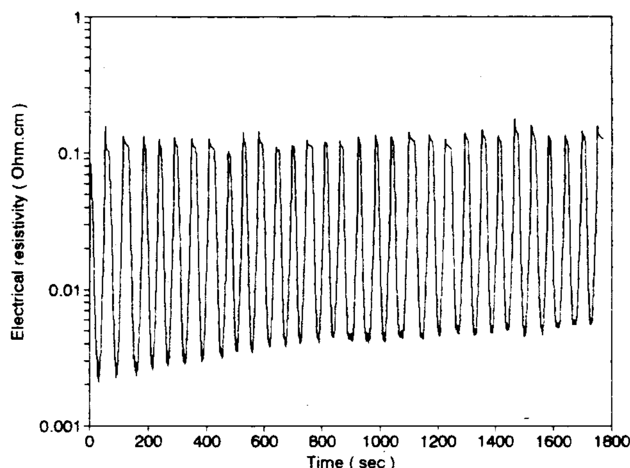

Fig. 3. Variation of the z-axis resistivity for the overall film (with 71 wt.% Ni) during cyclic compression between stresses of 0 and 1.40 MPa.

Figure 2 shows scanning electron microscope (SEM) photographs of the z-axis film viewed along the z axis (Fig. 2a) and viewed perpendicular to the z-axis (i.e., edge view, Fig. 2b). Figure 2a shows the random two-dimensional distribution of the Ni particles. Figure 2b shows the protrusion of the Ni particles out of both surfaces of the film and that each conducting path consists of a single Ni particle. The thickness of the polymer part of the film was ~50 μm, while that of an Ni particle was ~70 μm.

Figure 3 shows the variation of the z-axis resistivity (for the overall film with 71 wt.% Ni) during cyclic compressive loading between stresses of 0 and 1.40 MPa. The reversibility of the resistivity changes shows that the film was resilient, so that the film can serve for numerous times as a separable connector. Plots similar to Fig. 3 were also obtained for films with 60 and 67 wt.% Ni. The extent of compression (i.e., the shrinkage during compression) at 1.4 MPa in the z-axis was 14 μm for a film (i.e., 7 μm for each side of a film), as shown in Fig. 4 for the film with 60 wt.% Ni. This means that only the Ni particles (not the polymer) deformed during compression.

The z-axis resistivity of all the films did not change during long-term application of a constant pressure, as shown in Fig. 5 for the film with 71 wt.% Ni at a pressure of 1.39 MPa. The z-axis compressive stress at a constant z-axis compressive strain also did not change (except for the slight variation due to the temperature variation during each day of the three days of measurement), as shown in Fig. 6 for the film with 71 wt.% Ni at a constant strain of 0.93%. Figure

6 indicates the absence of stress relaxation and is consistent with Fig. 5 and the fact that the polymer was not deformed.

Z-Axis Adhesive

The resistivity of the z-axis adhesive film is shown in Table II. The trends with respect to the Ni content is similar to those of the standalone film (Table I). However, the conducting path density is higher, the pitch is lower, the z-axis resistance within one path is lower (compared at zero pressure) and the z-axis resistivity is higher for the adhesive film than the standalone film.

DISCUSSION

Due to the proprietary nature of the z-axis film technology developed in various companies,[1–5,7–16] comparison of the processing, structure and properties of films from different sources is difficult. The only published comparative study is Ref. 17, which covers resilient films from AT&T, Fujipolymer Inc. and Japan Synthetic Rubber Company. Table III shows the comparison among these films, the films of other papers,[6–9,13,14,18] and ours. This comparison indicates that our standalone film is outstandingly low in the z-axis resistance and is among the lowest in film thickness; our adhesive film is the lowest in pitch and thickness, but relatively high in the z-axis resistance.

The overall z-axis resistance (Table III) is high for our adhesive film, due to the low nickel volume fraction and the low fraction of nickel particles that are large enough to serve as z-axis conduction paths. Compared to the z-axis adhesive film of 3M Corp.,[13,14] our z-axis adhesive film is high in the overall z-axis resistance, partly due to the particles in our film being not compressed, whereas those of the 3M film were compressed into an oval shape. However, our film has the advantage of requiring no heat or pressure for curing, whereas the 3M film requires 180–190°C and 1.9 MPa for curing. In spite of the high overall z-axis resistance of our adhesive film, the z-axis resistance within one path (Table II) is not high compared to our standalone film (Table III). Compared to our standalone film, our adhesive film is lower in pitch and in cost. The low pitch and consequent high conducting path density are due to the smaller size of the

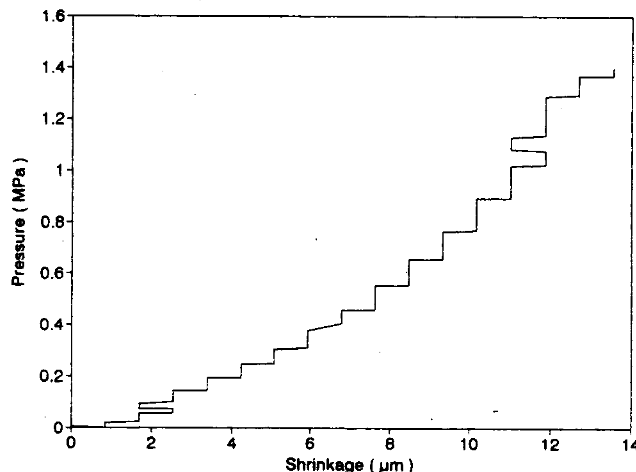

Fig. 4. Z-axis shrinkage of film (with 71 wt.% Ni) during compression from 0 to 1.40 MPa.

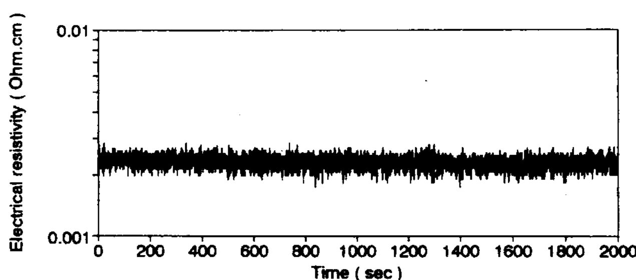

Fig. 5. Variation of the z-axis resistivity for the overall film (with 71 wt.% Ni) during compression at a constant stress of 1.39 MPa.

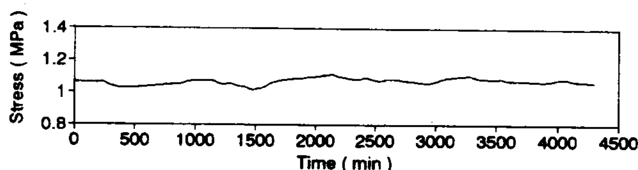

Fig. 6. Variation of the stress during compression at a constant strain of 0.93% for the film with 71 wt.% Ni.

Table II. Electrical Resistivity in the Z-Axis of Z-Axis Conductor Adhesive Films

Wt.% Ni	60	67	71
Vol.% Ni*	17	22	24
Conducting path density (cm^{-2})	1.7×10^{-1}	2.2×10^{-1}	2.5×10^{-1}
Pitch+ (μm)	76	67	64
Z-axis resistance within one path (Ω)	53	55	52
Z-axis resistivity[1] (Ω · cm)	8.7×10^{-1}	7.1×10^{-1}	6.1×10
Effective conducting path diameter** (μm)	0.24	0.24	0.24

*Calculated from wt.% Ni using densities of Ni and PVDF.
+Average distance between centers of adjacent paths.
[1]For overall film.
**Calculated from the z-axis resistance within one path by assuming that each path is a nickel cylinder.

Table III. Comparison of Z-Axis Conductor Films from Different Sources

Source	Grade Designation	Thickness (μm)	Pitch (μm)	Z-Axis Resistance (mΩ) for an Area of 1.4×10^5 μm²
AT&T*	ECPI (coarse-pitch)	250–265	> 250	30 at 0.69 MPa
	ECPl (fine-pitch)	250–265	> 250	120 at 0.69 MPa
Fujipolymer*	FP-R	500	100	115 at 0.69 MPa
	FR-WSS	500	250	> 100 at 0.69 MPa
Japan Synthetic Rubber*	PCR 303-C	250–1000	> 250	110 at 0.69 MPa
Ref. 6	25 vol.% copper coated polymer particles	50–100	150	11,000 at 0.04 MPa
Ref. 9	Selectively metalized PTFE membrane	178	375	31.8 at 0.34 MPa 12.7 at 0.68 MPa
This work (standalone film)	71 wt.% Ni	70	125	11.5 at 1.39 MPa 80 at 0.037 MPa
This work (adhesive film)	71 wt.% Ni	30	64	130
Ref. 13, 14 (adhesive film)	3M ZAF	50	150	< 10
Ref. 18 (adhesive film)	Merix ABC	50-125	1500	—

*From Ref. 17

nickel particles in the adhesive film than in the standalone film. Compared to the z-axis adhesive film of Merix Corp.,[18] our z-axis adhesive film is very low in pitch.

The combination of low pitch, low z-axis resistance and small thickness makes our standalone film highly attractive. The low z-axis connection resistance of our standalone film is due to the fact that each path comprises one nickel particle of diameter 70 μm, in contrast to previous technologies that use either multiple particles or a single metal coated polymer particle for each path. A further advantage of our film is the low processing cost, which stems from the fact that magnetic alignment, metal coating (to form the conducting particles) and precious metals (such as gold) are all not needed, in contrast to the processing of other z-axis films. The resilience stems from the resilience of the nickel particles, in contrast to previous separable z-axis films,[1,2,6–8] the resilience of which stems from that of the polymer (an elastomer). A consequence of the absence of polymer deformation is the absence of stress relaxation.

CONCLUSION

This paper provides z-axis conductor films in standalone and adhesive forms for use as electrical interconnections. They are polymer-matrix composites with one nickel particle per conducting path. The matrix materials are PVDF and epoxy for the standalone and adhesive films respectively. By using 71 wt.% Ni (33 vol.% Ni), a resilient standalone film with a pitch of 125 μm, a z-axis connection resistance of 2.4 Ω and a z-axis resistivity of 2.3×10^{-3} Ω · cm for the overall film at a z-axis pressure of 1.39 MPa was attained. Each nickel particle protruded from both sides of the standalone film, such that the film was ~50 μm thick in the polymer portions and ~70 μm thick at the nickel particles. The films were made by hot pressing a mixture of nickel and PVDF particles between two copper foils, the ductility of which allowed the nickel particles to protrude. Film deformation during compression involved nickel particle deformation and not polymer deformation, so the films exhibited no stress relaxation. By using 71 wt.% Ni (24 vol.%Ni), an adhesive film with a pitch of 64 μm, a z-axis connection resistance of 52 Ω and a z-axis resistivity of 0.61 Ω · cm for the overall film at zero pressure was attained.

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