ELECTROLESS COPPER DEPOSITION ON LOW-TEMPERATURE SILVER THICK FILMS

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A disruptive method of manufacturing an electronic module by additive processes without using soldering raises several challenges. One of them is the current capability improvement of the interconnection structure made by composite materials through an economic method. Several electrically conductive materials with similar volume resistivity have been studied in order to investigate the effect of the electroless copper plating over the electrical resistance of a test pattern. The presence of silver in these materials facilitated the obtaining of a simple plating process. The quality of the copper deposition was investigated through electrical measurements and microstructural analysis.

Keywords: electroless copper plating, solderless assembly for electronics, electrically conductive adhesive, volume resistivity, thick film

1. Introduction

In recent decades, the electronic packaging engineering community has been increasingly looking for an alternative to soldering. Especially after the entry into force of the RoHS EU Directive with its increased thermal stress required by most lead-free alloys, the experts say that almost 70% of defects in electronic products are due to the joints and other related issues to the soldering process [1]. Moreover, in thin-film solar cells, lead-free soldering becomes unsuitable for its increased heat input. The progress in electrically conductive adhesives (ECA) led to the idea of replacing the solder alloy on the surface mount technology lines [2,3,4], and photovoltaic modules assembly [5]. Although this was the most convenient solution for solderless assembly for electronics [6], no massive shift to ECA assembly was yet observed. ECAs are mainly used in applications where the risks of mechanical and thermal cracking are very high, or when there are

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concerns about damaging heat-sensitive components on the printed circuit board (PCB) during soldering. However, the most important approach in the field is the additive method [7]. Hensoldt [8] together with Nano Dimension [9], has announced the use of 3D printing for building the first ten-layer 3D PCB. The board was manufactured utilizing newly developed dielectric polymer ink and conductive ink, but the components must be still attached by soldering.

Verdant Electronics (USA) [10] launched a more disruptive approach: the Occam Process [11]. According to this concept, two technological processes - the manufacture of the PCB and the assembly of components on the board, sometimes made in different factories located at appreciable distances - merge into one in which the entire module is realized by additive processes at a single location. In a model for manufacturing an electronic module based on the Occam concept, the problem of creating the interconnection structure is the most challenging. To avoid the deposition of electrolytic copper, which is energy consuming and further involves an etching operation, it is proposed to deposit the tracks of a composite electrically conductive material. The deposition should be performed by using cheaper printing methods. There are many such materials on the market in the form of paste or ink, but all have a higher volume resistivity than technical copper [12], which limits the field of application. There are several options for increasing current capability that may be considered, as follow:

i) ***the use of nanoparticle conductive materials****.*

Advances in technology have made it possible to obtain new conductive materials based on nanoparticles. The use of thin-film or coating instead of conductive metal foil is seen as a future alternative in the electronic and optoelectronic industry [13]. High conductivity ECAs at low filler content was achieved by fully utilizing the synergistic effect of nano- and micro-scale fillers. Thus, nano silver-coated copper micron flakes prepared by an electroless plating method combined with Ag nanowires made by a simple one-step polyol method have been proved to significantly reduce the bulk resistivity of ECAs. As result, the resistivity of ECA was dropped down to 9.42E-07 Ω m when the filler content was only 60 wt% [14]. A recent achievement is the AgCite^{TM} 90072 silver nanoparticle conductive ink from Nano Dimension [9] which has a conductivity of 3.15E06 to 2.52E07 S/m @ 20 °C, meaning a volume resistivity of 3.17E-07 to 3.97E-08 Ω m, depending on printing and sintering conditions. However, access to these materials is tough and their cost is prohibitive (more than 12 €/ml) for common applications. Ink deposition is also a challenge for existing technology in small and medium enterprises.

ii) the selection of proper curing parameters for the ECA.

The research revealed the influence of curing parameters on the electrical properties of some conductive adhesives. The volume resistivity of a typical

isotropic conductive adhesive (ICA), composed of an epoxy-based binder and a filler with small silver flakes showed a significant reduction of an order of magnitude when cured in a single step process at 250 °C versus 180 °C [15].

A multi-step method also decreased the bulk resistivity of ICAs though they were cured for the same periods, it was further found that their electrical resistance continued to decrease during the subsequent post-heating treatment processes [16]. Generally, the manufacturers of electrically conductive composite materials offer the volume resistivity variation in the datasheet depending on the curing parameters (see Table 1) [17,18].

Table 1

Volume resistivity variation *vs* curing parameters for three materials [17,18]

Material, manufacturer	Curing parameters	Volume resistivity, [Ω m]
Compound Silver 402 (Loctite)	15 min @ 120 °C	2.00E-06
	30 min @ 100 °C	4.00E-05
	2 h @ 65 °C	6.00E-05
	24 h @ 25°C	3.00E-04
Elecolit 414 (Panacol-Elosol GmbH)	16 min @ 70 °C	5.00E-06
	5 min @ 150 °C	5.00E-07

As it can be observed, a curing profile with a shorter time and a higher temperature is favorable to lower the volume resistivity. It is a good time interval for the yield, but higher temperatures could affect some types of substrates (e.g., change of color of the epoxy resin at 150 °C). Compared to copper (1.68E-08 Ω m) or silver (1.59E-08 Ω m) [19], the volume resistivity values are still high.

iii) adding a layer of electroless copper on the conductive traces.

To reduce the waste, copper should be added only to the conductive traces. Therefore, methods like sputtering, physical vapor deposition, electroplating, or chemical vapor deposition are not adequate. Better methods for enhancing the electrical properties of silver ink are electrolyte sintering, photonic sintering, and electroless copper plating (ECP) [20]. As known, ECP is a chemical process that deposits a thin and even layer of copper, generally, without using an electric current passing through the bath. Thus, copper can be plated both on conductive and non-conductive solid surfaces. Selectivity is conferred by the presence of catalytic metal in the composition of the conductive material. Good electrical resistivity (2.30E-08 Ω m @ 3.6 μm maximum effective copper coating thickness) can be obtained even by a facile method [21]. Although a mature process since being known for a long time, progresses in copper electroless plating is still noticed [22]. Among them, the use of lasers has led to several new technologies.

The ability to control the laser ray was used to make selective ECP creating conductive traces on a flexible board of polymer resin [23]. Here, the catalytic surface was obtained by laser irradiation on the flexible board previously

immersed in an $AgNO_3$ solution. The report emphasizes uniform copper deposition and good conductivity properties. However, the solution involves a serial treatment of the routes which leads to a longer process duration (40 min). In traditional PCB manufacturing, ECP is used to make plated-through holes and vias, as well as external surfaces.

The thickness of the plating is typically 0.5÷2.5 μm both in the holes and on the surfaces. Next to the ECP, the copper thickness is increased by other methods using electric current. ECP is used also in plating 3D polymer microstructures to obtain metallization of micro-electromechanical systems [24], and wearable electronics [25].

The goal of the present work is the investigation of the possible increase of the current capability of the conductive traces by ECP. Following Ohm's law, this will be done indirectly, by measuring the electrical resistance of some test tracks. There will be a quantitative and qualitative assessment of Cu deposits made by electrical measurements and microstructural investigations. The expected benefits of Cu electroless plating are due to the following considerations: i) Cu has a very good electrical conductivity; ii) Cu deposition allows (or improves) solderability; iii) it is a uniform and conformal deposition; iv) the technique is simple, low cost, and easy to implement.

2. Materials and Method

To deposit copper on a limited number of test specimens, a mini ECP line was prepared. The source of copper used is the copper sulfate pentahydrate ($CuSO_4$ $5H_2O$), while the reduction agent was chosen formaldehyde (HCHO) instead of glyoxylic acid. As known, glyoxylic acid is very sensitive to temperature and concentration making it difficult the control the plating solution [26]. The plating temperature of the solution was set to 40 °C, 5 Celsius degrees lower than other lower temperature approaches [27,28]. The low temperature was chosen because it was reported that the resistivity of the deposits is lowest when using a formaldehyde high concentration agent [29]. On the other hand, it was reported that at low temperature the copper deposits present voids in the over-etched cross-sections and solid structures of the surface morphology [30].

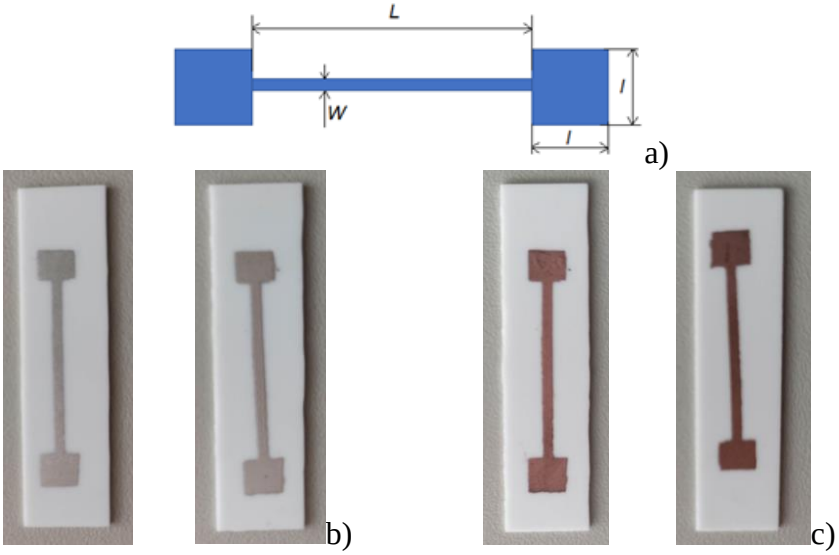
Three of the electrically conductive materials with the best volume resistivity/sheet resistance values were selected for investigation: **M1** - Elecolit® 414 (from Panacol-Elosol GmbH); **M2** - Bectron® CP 6662 (from ELANTAS Europe GmbH); **M3** - SW180 (Tatsuta Electric Wire & Cable). There are different types from different manufacturers, but all have silver as a filler (see Table 2) [18,31,32].

Table 2

Main properties of the materials used in the experiment [18,31, 32]

Properties	Electrically conductive materials		
	M1 Elecolit 414	**M2** Bectron® CP 6662	**M3** SW180
Type	Conductive ink	Conductive ink	Conductive paste
Filler	Silver, flakes	Silver; flakes	Silver coated Cu, balls
Filler/solids weight [%]	87	73	90
Binder	Polyester	Thermoplastic resin	Epoxy
Curing temp. [°C]	70 / 125 / 150	120	130 / 160
Vol. resistivity [Ω m]	5,00E-07, PE-Norm 040	< 2.54E-07	5.30E-07 @ 200 µm width

Fig. 1a) shows the test vehicles consisted of a test structure pattern built on Al_2O_3 96% substrates, Figure 2b) shows the samples on which the deposits were performed before ECP, and Figure 2c) after ECP.



a) b) c)

Fig.1. a) Test structure (L x W x l = 20 x 1 x 4 mm); deposits were performed before ECP (b), and after ECP (c).

To evaluate the effect of ECP on the electrical conductivity of the composite material two methods were used.

I. 4-Point Measurements

The volume resistivity is indirectly determined by measuring the electrical resistance of a sample of material of known geometry. Because there are involved very low values of resistance, the "4-Point Measurement" method was chosen. The method is recognized for avoiding the measuring errors caused by the

resistance of the wires between the sample and the instrument [33]. The measurements were performed with Keithley 2460 SourceMeter Unit instrument.

II. Electrical Resistance Measurement

The effect of the copper electroless plating over the electrical conductivity property was studied by comparative measurements on test vehicles performed before and after copper deposition.

The experimentation took place by following the steps:

a) The deposition of the conductive material on the two substrates by screen printing method followed by thermal treatment (15 min @ 130 °C).

b) The measurement of the electrical resistance of the conductive deposits from the test vehicles.

c) The copper electroless plating process (or autocatalytic metal deposition) on half of the test vehicles (15 min @ 40 °C).

Electroless plating (or autocatalytic metal deposition) is a redox process where a metal ion is simultaneously reduced by a reducing agent at a catalytic surface:

$$2CH_2O + 4OH^- \rightarrow 2CHO_2^- + 2H_2O + H_2 + 2e^- \quad (1)$$

$$Cu^{2+} + 2e^- \rightarrow Cu \quad (2)$$

After a catalytic center is coated with Cu, the copper itself further catalyzes the process. Reaction kinetics is influenced by the pH of the solution, temperature, bath composition, etc. Catalytic surfaces can be Pd, Pt, Au, Ag, Cu, Al, etc. Metals deposited by electroless plating can be Ag, Au, Cu, Ni, Co, etc. Reducing agents can be Sodium tetrahydroborate ($NaBH_4$), Hydrazine (H_2N-NH_2), Sodium hypophosphite (NaH_2PO_2), Formaldehyde (CH_2O), etc.

Since the catalytic metal is the same for all the materials (Ag), and the same process was used, it is supposed the copper layer has the same thickness for all the samples.

Thus, the experimental conditions for electroless deposits on the silver layer (alumina and glass epoxy) were as follows:

- Reducing agent: CH_2O;
- Cu source: Copper sulfate pentahydrate ($CuSO_4 \times 5\ H_2O$);
- pH: 12;
- Temperature: 40 °C;
- Time: 15 min.

d) The measurement of the electrical resistance of the conductive deposits with an electroless copper layer from the corresponding test vehicles.

III. SEM-EDS Microscopy

For scanning electron microscopy (SEM), we used a Phenom ProX SEM, equipped with an X-ray spectrometer with EDS energy dispersion.

In Fig. 3 the results of the SEM-EDS (combined BSD Topo mode and elements distributions) investigations are presented for the sample with Al_2O_3 substrate, on which the M1 (Elecolit 414) paste was deposited. Ag flakes were distributed relatively evenly on the alumina plate, being randomly oriented. The deposition shows microzones with a tendency to superposition, accompanied by a local non-coverage of the support plate. According to EDS mapping, the wt% of identified elements is as follows: 10%C; 85%Ag; 4%O; 0.4%Al; 0.6%AE.

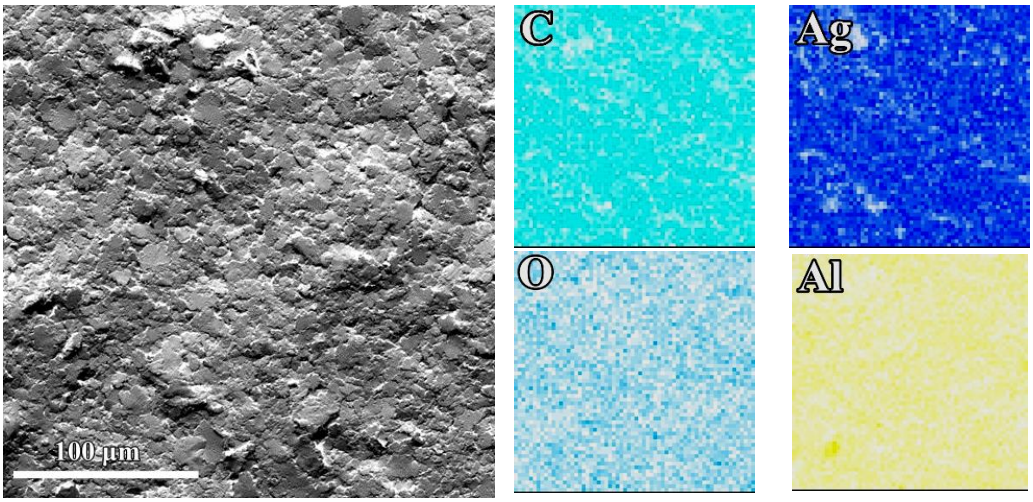


Fig. 3 SEM-EDS (combined BSD Topo mode and elements distributions) microscopy of the sample with Al_2O_3 substrate, on which the M1 (Elecolit 414) paste was deposited.

In Fig. 4 the results of the SEM-EDS (combined BSD Topo mode and elements distributions) investigations are presented for the sample with Al_2O_3 substrate, on which the M1 (Elecolit 414) paste was deposited and, after ECP. Cu follows the morphology of the Ag flakes of the deposited intermediate layer being plated relatively unevenly, in some microzones missing (compare EDS orange mapping of Cu with the blue of Ag). Due to the very thin layer of Cu plated, the proportion of Ag is maintained at high values (approx. 42%), compared to 38%Cu. According to EDS mapping, oxygen is present in a proportion of 10%, and Al of about 1%, the rest being AE.

In Fig. 5 the results of the SEM-EDS (combined BSD Topo mode and elements distributions) investigations are presented for the sample with Al_2O_3 substrate, on which the M2 (Bectron® CP 6662) paste was deposited. Ag flakes were distributed relatively unevenly on the alumina plate and their orientation in

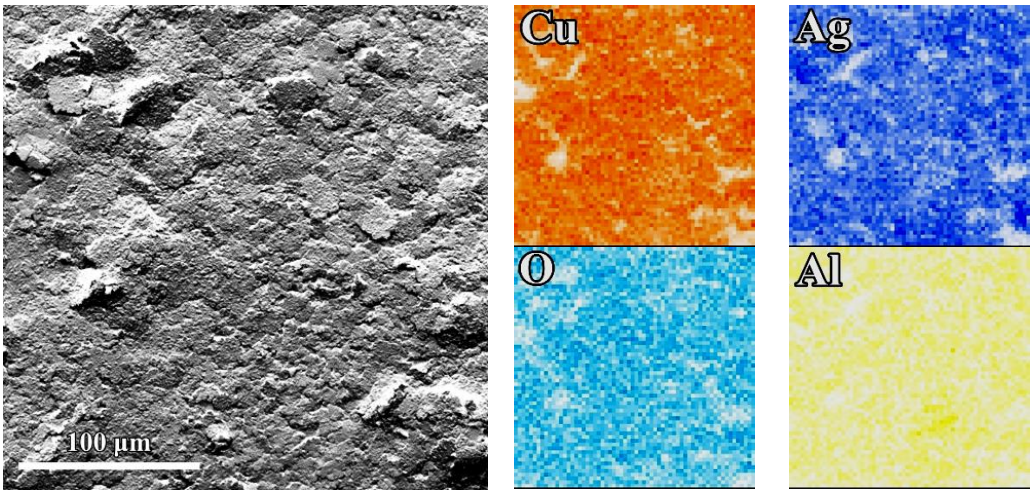


Fig. 4 SEM-EDS (combined BSD Topo mode and elements distributions) microscopy of of the sample with Al_2O_3 substrate, on which the M1 (Elecolit 414) paste was deposited and, after ECP.

the deposit plane is random; the deposition shows microzones with a tendency to agglomerate, accompanied by a local non-coverage of the support plate. According to EDS mapping, the wt% of identified elements is as follows: 11%C; 79%Ag; 8%O; 0.3%Al; 1.7%AE.

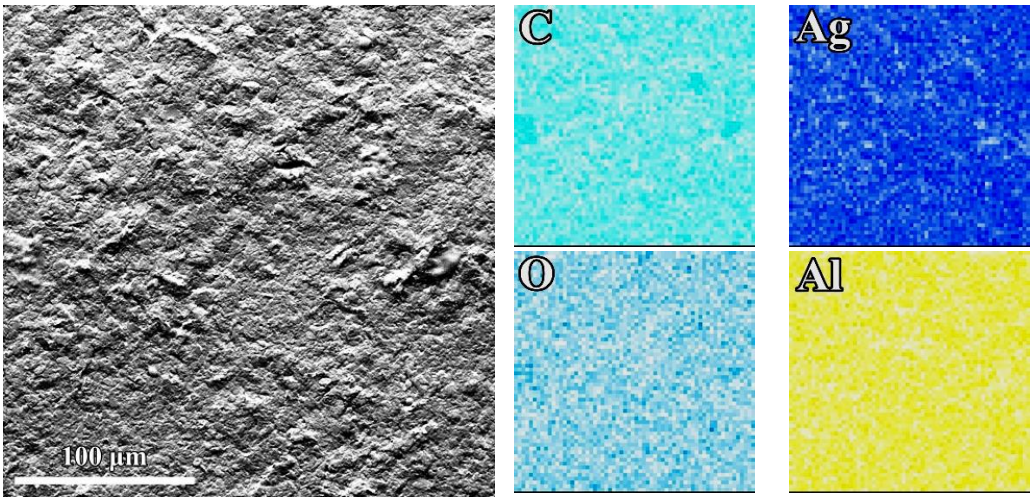


Fig. 5 SEM-EDS (BSD full mode and elements distributions) microscopy of of the sample with Al_2O_3 substrate, on which the M2 (Bectron® CP 6662) paste was deposited.

In Fig. 6 the results of the SEM-EDS (combined BSD Topo mode and elements distributions) investigations are presented for the sample with Al_2O_3 substrate, on which the M2 (Bectron® CP 6662) paste and after ECP. Cu follows the morphology of the Ag flakes of the deposited intermediate layer being plated

relatively uniform (compare EDS orange mapping of Cu with the blue of Ag). Due to the very thin layer of Cu plated, the proportion of Ag is maintained at values of about 3.3%), compared to 83%Cu. According to EDS mapping, the wt% of identified elements is as follows: 5.6%C (free of mapping); 4.2%O; 1.3%Al; 2.6%AE.

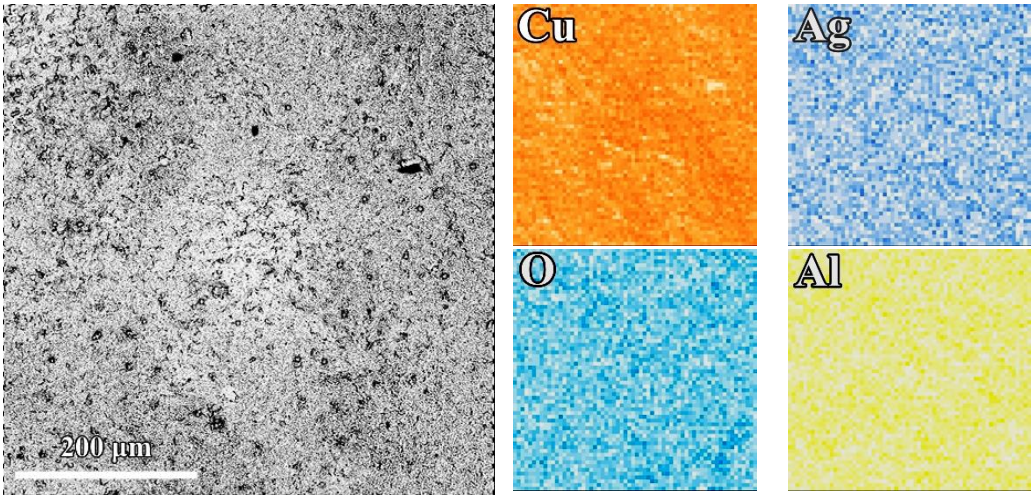


Fig. 6 SEM-EDS (combined map BSD, Topo B mode and elements distributions) microscopy of of the sample with Al_2O_3 substrate, on which the M2 (Bectron® CP 6662) paste was deposited and after ECP.

In Fig. 7 the results of the SEM-EDS (combined BSD Topo mode and elements distributions) investigations are presented.

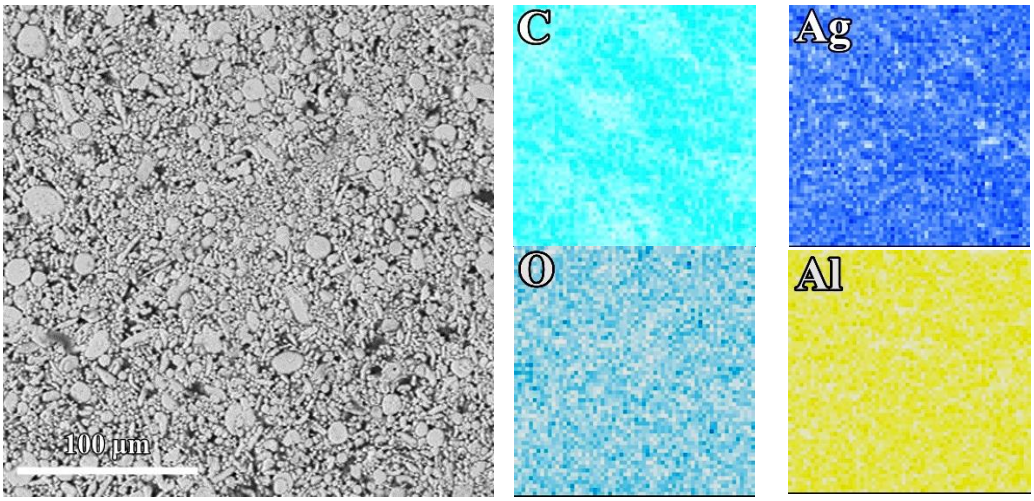


Fig. 7 SEM-EDS (BSD full mode and elements distributions) microscopy of of the sample with Al_2O_3 substrate, on which the M3 (Tatsuta SW 180) paste was deposited.

The sample with Al_2O_3 substrate, on which the M3 (Tatsuta SW 180) paste was deposited Ag spheroids was distributed relatively evenly on the alumina plate, being randomly oriented. The deposition shows microzones with a tendency to superposition, accompanied by a local non-coverage of the support plate (black points on the SEM image). According to EDS mapping, the wt% of identified elements is as follows: 20%C; 62.8%Ag; 11.7%O; 1.4%Al; 4.1%AE.

In Fig. 8 the results of the SEM-EDS (combined BSD Topo mode and elements distributions) investigations are presented for the sample with Al_2O_3 substrate, on which the M3 (Tatsuta SW 180) paste was deposited and after ECP. Cu follows the morphology of the Ag spheroids of the deposited intermediate layer, following a slight band-oriented distribution, probably according to the morphology of the support, being plated relatively evenly (compare EDS orange mapping of Cu with the blue of Ag). Due to the very thin layer of Cu plated, the proportion of Ag is maintained at the values of about 11.2%), compared to 60.7%Cu.

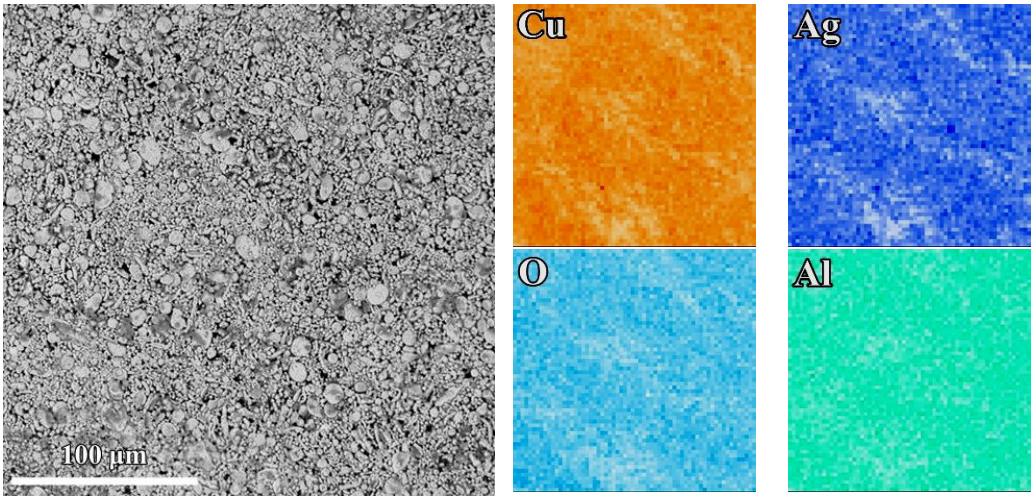


Fig. 8 SEM-EDS (combined map BSD, Topo B mode and elements distributions) microscopy of of the sample with Al_2O_3 substrate, on which the M3 (Tatsuta SW 180) paste was deposited and after ECP.

According to EDS mapping, oxygen is present in a proportion of 6.3%, and Al of about 0.48%, the rest being AE.

As a general remark, the EDS mappings / measurements are affected by the very small thickness of the layers, the values for copper and silver, interfering both with each other and with the elements that make up the organic part of the electrically conductive materials studied.

3. Results and Discussion

The values of the electrical resistance measurements (4-Points Measurements, with Keithley 2460) are presented in Table 3.

In terms of volume resistivity, the materials M1 and M3 are very similar, which could be explained by similar filler weight content, but the initial measurement of the electrical resistance showed large differences.

Table 3

Electrical resistance measurements

Material	Substrate	R, [Ω]		Electrical resistance reduction, [%]
		Before ECP	After ECP	
M1	Al_2O_3	1.95	0.99	49.2
M2	Al_2O_3	1.26	0.65	48.4
M3	Al_2O_3	0.66	0.36	45.5

This may be due to drying/curing conditions that followed the printing of the routes. M3 conductive paste samples presented the lowest electrical resistance before and after the electroless copper plating, which was to be expected given that it has the highest proportion of metal filler among the three materials (90%). The electrical resistance reduction due to the thin copper layer is obvious in all the samples. The support of the conductive traces does not seem to influence the measurements of the resistance.

Rearranging the data from Table 3 in descending order of electrical resistance it can be observed that the reduction of the resistance after ECP is more effective for higher initial values of the resistance before ECP. For lower initial values, the electrical resistance value has no longer a very large reduction. Further reduction could be obtained by increasing the thickness of the copper layer.

4. Conclusions

This paper described the recent development, research works, and applications of conductive adhesives as one of the promising lead-free alternatives for electronic packaging and interconnection applications.

Conductive adhesive materials have evolved to meet the higher electrical/mechanical/thermal performance, low-temperature process, and strong adhesion/reliability requirements for electronic packaging modules and assemblies.

Improvements in the resistance value are evident for all three conductive materials based on silver filler due to copper’s very good electrical conductivity.

Consequently, the ECP could be a solution for increasing the current capability of traces made by electrically conductive pastes or inks based on silver.

The technique is simple, the cost is low, and the process is easy to implement. It is a uniform and conformal deposition.

Regarding the morphology of the copper layers, the best conformal deposition was obtained with the Bectron 6662 samples.

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