

Original research article

Investigation of poly (2-methyl imidazole co 1,4-butanediol diglycidyl ether) as a leveler for blind hole copper plating

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Abstract: The current synthetic leveler agents are complex to operate, troublesome to purify, and low to pore filling rates. Herein, we report a new, simple, green, and inexpensive method for the synthesis of leveler agents and study their properties systematically. In acidic copper sulfate plating solutions, including polyethylene glycol (PEG), sodium 3,3'-dithiodipropene sulfonate (SPS), and chloride ions, the relation between leveler agents (dimethylimidazole and 1,4-butanediol diglycidyl ether) and the filling of blind holes have been investigated. The synthesized leveler agent was structurally characterized by infrared spectroscopy and gel chromatography tests. The plating containing the leveler agent passed plating tests as well as reliability tests and met PCB production requirements. The electrochemical behavior of the leveler agent was investigated using electrochemical tests. The X-ray diffractometer (XRD) was used to observe the differences in crystal orientation on the surface of the plated layers.

Keywords: copper plating; blind holes; leveler; poly (2-methylimidazole co 1,4-butanediol diglycidyl ether)

Received: 6 July 2023; Accepted: 7 August 2023; Available online: 9 September 2023

1. Introduction

Printed circuit boards (PCB) are an indispensable part of our daily life. As electronic devices are now moving toward lighter, thinner, and smaller, printed circuit boards are also moving toward multilayers and higher densities. The multilayer design of PCB can not only improve the space utilization of PCB, but also avoid the interference of electrical signals and noise, thus satisfying the needs of various complex and special applications. Due to the good heat dissipation properties of copper, copper-plated blind-holes can improve the heat dissipation performance of PCBs. Moreover, blind holes are a convenient path to connect each layer of the PCB without running through the entire board, which is conducive to improving the efficiency of the PCB^[1-3].

However, in order to satisfy the requirements of current electronic devices, copper-plated blind holes need to be perfectly filled and free of cracks and gaps, which requires blind holes to be super-filled or top-down filled. Only when the bottom rate of copper deposition in the blind hole is much greater than that of at the mouth and surface of the hole when plating the blind hole, ultimately, the blind holes will be perfectly filled. Owing to the acidic environment provided by sulfate copper plating system, copper ions is easier to deposit to form a layer of copper plating with strong adhesion and high brightness on the surface of the plated parts. Moreover, sulfate system is favored by the plating industry because of its low cost of raw materials. Other

copper plating systems such as chloride, oxalate, borate, etc., are blocked by higher costs, poorer operability, environmental issues, etc.

To satisfy the need for super-filling, in the industry, acidic copper sulfate is a kind of common system with accelerators^[4], inhibitors, and leveler agents as additives. In the industry, acidic copper sulfate is a kind of common system with accelerators, inhibitors, and leveler agents as additives. Accelerators are mainly organic compounds containing sulfur groups, which can be adsorbed on the electrode surface during plating, thus refining the copper crystal structure and promoting the generation of copper nuclei. Furthermore, accelerators mainly combine with chloride ions to depolarize and increase the current density of the cathode^[5-8]. In recent years, accelerators such as SPS and MPS have been widely studied and used^[9-11]. Inhibitors, like polyether and so on, are mainly adsorbed on the surface of the plated part to prevent the diffusion of Cu^{2+} and inhibit the deposition of copper during the plating process^[12-14]. When both accelerators and inhibitors are presented in a system, there will exist adsorption competition, and the presence of leveling agents is a key factor in stabilizing the system^[15-17]. Most leveler agents are nitrogen-containing organics^[18,19]. During the copper plating process, nitrogen-containing organic compounds can form stable complexes with copper ions, which helps copper ion deposition. Moreover, nitrogen-containing organic compounds can be selectively adsorbed on the substrate to improve plating uniformity. What's more, nitrogen-containing organic compounds can be stabilized in acid sulfate plating solution and are not easy to decompose. Therefore, nitrogen-containing organic compounds are a good choice as leveling agents. In PCB processing, in addition to nitrogen-containing organic compounds, the following compounds are also suitable to use as leveling agents: (1) mercapto compounds such as mercapto alcohols, mercapto ketones, etc.; (2) quaternary ammonium compounds such as benzyltrimethylammonium chloride; (3) certain organophosphorus compounds such as phosphate esters. Typical organic components of leveling agents include the following: (1) mercapto components; (2) quaternary ammonium salt components; (3) nitrogen-containing heterocyclic components; (4) double bonds and conjugated systems. These organic components play a key role in the leveling agent for copper electroplating. They regulate the growth process of the plated layer through chemical interaction with copper ions to form a uniform and smooth surface. To the best of our knowledge, currently reported dye-based leveler agents are JGB^[20], DB^[21], ABPV^[22], etc., and non-dye heterocyclic leveler agents are 2-amino-4-methylbenzothiazole (AMBT)^[23] and 2-methylpyridine (2-MP)^[24,25], etc. The role of leveler agents is (1) to keep synergistically with inhibitors to inhibit the deposition of copper on the surface of the plated part; (2) to improve the current density and increase the hole-filling rate, thus avoiding the phenomenon of cavitation due to early sealing during the hole filling process^[26-31].

The important role of leveler agents in super-filling holes has been demonstrated by researchers^[20,32-34]. For example, Janus Green B (JGB) is an excellent leveler agent for electroplated copper, which not only enhances the filling rate of blind holes but also reduces the bulging of the filled holes. Lee et al.^[35] synthesized a leveling agent containing two quaternary ammonium groups and hexamethylene glycol and found that the leveling agent could achieve seamless and nonporous filling of blind holes. What's more, TCA^[23] and DMP^[36] have also been reported as potential new leveling agents. Although the research on leveling agents has achieved certain results, owing to the top secret, expensive, and environmentally unfriendly of most leveler agents, the development of leveler agents is a time-consuming and laborious task^[26]. Therefore, it is urgent to develop a leveler agent that is easy to synthesize, green, and inexpensive to speed up the commercialization process^[37-39].

Herein, we reported a green, simple, and inexpensive method to synthesize a kind of leveler agent for the first time. Specifically, we synthesize a new type of super filling leveler agent using dimethylimidazole and 1,4-butanediol diglycidyl ether and characterize its organic structure, investigate the performance of the leveler agent in plating and study its electrochemical behavior systematically. In comparison with other leveling agent

related literature, the synthesis and purification methods used in this paper are simple and reduce the cost of the synthesis process. Moreover, this paper uses water as the solvent for synthesis instead of organic solvents, which is more environmentally friendly than other synthetic leveling agent methods. Furthermore, the phase structure and contact angle of the copper coating were tested, and the thermal shock test was carried out. Above experimental results show that the plating solution formula of the leveler agent synthesized by this method can fully meet the needs of PCB applications.

2. Experimental details

2.1. Synthesis of leveler agents

Initially, a predetermined amount of 2-methylimidazole is placed into a 100 mL round-bottom flask, after which a specific volume of water is added to dissolve the 2-methylimidazole. The 2-methylimidazole solution is then heated to a specified temperature, followed by the gradual addition of 1,4-butanediol diglycidyl ether with continuous stirring for 30 min. Subsequently, the temperature of the mixed solution is raised to 90 degrees Celsius, and the reaction is allowed to proceed for 18 h. After the completion of the reaction, the pH of the product is adjusted to 2–3. In comparison with other leveling agent related literature, the synthesis and purification methods used in this paper are simple and reduce the cost of the synthesis process. Moreover, this paper uses water as the solvent for synthesis instead of organic solvents, which is more environmentally friendly than other synthetic leveling agent methods.

The molecular structure of the reactants (2-methylimidazole and 1,4-butanediol diglycidyl ether) and the synthesis of poly (2-methylimidazole co-1,4-butadiene diglycidyl ester) are shown in **Figure 1**, where the synthesized product is codenamed ADT-90.

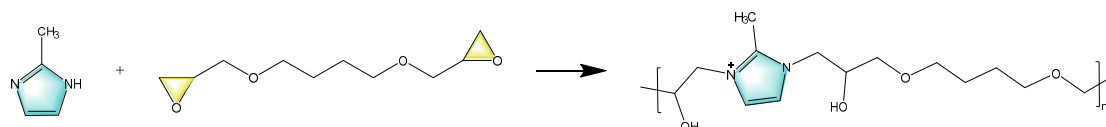


Figure 1. Molecular structure of the reactants (2-methylimidazole and 1,4-butanediol diglycidyl ether) and synthesis reaction of poly (2-methylimidazole co-1,4-butadiene diglycidyl ester).

2.2. Characterization of leveler agents

In order to analyze the molecular structure of the products, the products were characterized by Fourier transform infrared spectroscopy (FTIR), hydrogen nuclear magnetic resonance spectroscopy (^1H NMR), and gel permeation chromatography (GPC). FTIR measurements were conducted by Nicolet iS50R (THEMOR-FILSHER, USA). The spectra were obtained in the wave number range from 400 to 4000 cm^{-1} , testing 16 cumulative cycles. Nuclear magnetic resonance hydrogen spectroscopy (^1H NMR) is measured by the Bruker AVANCE III 400 MHz superconducting Fourier (Bruker, Switzerland). The gel permeation chromatography (GPC) test was performed on Waters 1525 to analyze the molecular weight of the product.

2.3. Electrochemical testing

In this paper, the electrochemical properties of additives were tested on the CHI 760e electrochemical workstation by cyclic voltammetry (CV), chrono-potentiometric (GM), and Tafel curve test methods. The working electrode (WE) is a 3 mm diameter platinum rotating disc electrode (Pt-RDE); the reference electrode is a saturated mercurous sulfate electrode (SMSE), and the counter electrode (CE) is a platinum wire. The electrolyte was a basic plating solution (VMS) of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (220 g/L), H_2SO_4 (60 g/L), and chloride ions (60 mg/L) at a test temperature of 25 $^\circ\text{C}$ (water bath constant temperature, electrochemical test temperature in

this paper is 25 °C unless otherwise stated). In this study, sodium 3,3'-dithiodipropene sulfonate (SPS) was used as the accelerator, polyethylene glycol (PEG, MW = 10,000) as the inhibitor, and the leveler agent was the synthetic ADT-90.

In this study, cyclic voltammetry (CV) was performed from -0.8 V to 0.8 V with a constant scan rate of 50 mV/s. Constant current testing (GM) was performed under a current density of 1 A/dm², and various additives were added continuously during the measurements. The hole-filling performance of the plating solution was evaluated according to the difference in final potential between the two speeds, $\Delta\eta = \eta_{100 \text{ rpm}} - \eta_{1000 \text{ rpm}}$ ^[40]. The high speed, i.e., 1000 rpm, simulated the deposition of copper in the orifice as well as on the surface, and the low speed, i.e., 100 rpm, simulated the deposition of copper in the blind hole. If the deposition rate within the inner part of the hole is faster than the deposition rate of copper on the surface, the additive can be seen to have a filling effect. The bigger the difference in voltage between the rate of deposition on the inner and the surface of the hole, the better the pore-filling effect^[41,42].

In this study, Tafel curves are tested in a 3.5% NaCl solution under the open circuit potential ± 200 mV, with a scanning rate of 5 mV/s. Based on the test results, the corrosion current and corrosion potential can be determined. The more positive the corrosion potential and the lower the corrosion current of the test samples, the better the corrosion resistance of the plated layer is.

2.4. Phase structure testing

The test plates were cut to $0.5 \text{ cm} \times 0.5 \text{ cm} \times 0.5 \text{ cm}$, and oxides were removed from the surface with dilute sulphuric acid before testing. An X-ray diffractometer (MiniFlex 600 Japan) was used to test the grain orientation of the plated copper surface in the 2θ range from 20° to 90° .

2.5. Contact angle test

The corrosion resistance of electroplated copper on the surface of the plating was measured and analyzed with a corrosion solution by a Data Physics Instruments GmbH-OCA100 contact Angle tester. By the hanging drop method, the corrosion removal liquid was dropped on the substrate, and the coating surface with a drop volume of $5 \mu\text{L}$ and three different locations of each sample were tested, and the average value was taken.

2.6. Copper electroplating experiments

Blind hole test plates (FR-4 copper clad plates) size: $5 \text{ cm} \times 12 \text{ cm}$, blind hole diameter $140\text{--}160 \mu\text{m}$, hole depth $70\text{--}80 \mu\text{m}$ after chemical copper deposition, flash plating to obtain a thickness of $4\text{--}6 \mu\text{m}$ blind hole test plates with copper plating in the hole. The plating solution is consisted consists of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (220 g/L), H_2SO_4 (60 g/L), chloride (60 mg/L), polyethylene glycol (MW = 10,000) (400 ppm), bis-(3-sulfopropyl) disulfide (1 ppm) and synthetic leveler agent (1 ppm). The pre-treatment process for the test plates is shown as follows: 1) Acid degreasing for 1 min to remove oil from the surface, followed by water washing for 1 min ; 2) micro-etching in a sulphuric acid-sodium persulphate system for 30 s , followed by water washing; 3) soaking in a 3%–5% mass fraction sulphuric acid solution for 1 min , followed by plating in a 1.5 L Harling bath. The electroplating experiment employs a phosphor-copper anode measuring $5 \text{ cm} \times 12 \text{ cm}$. Under a current density of 1.6 A/dm^2 , electroplating will be conducted for 45 min at 25°C . Finally, the cross-section of the blind holes was observed through a metallographic microscope (MD50-B), and the plating effect was evaluated by calculating the filling rate of the blind holes, as shown in **Figure 2**, where the filling rate $F = (B/A) \times 100\%$, A is the thickness of the plating on the plate surface to the bottom of the blind hole, B is the thickness of the most concave part of the hole surface to the bottom of the blind hole, and C is the thickness of the plating during the filling process of the blind holes.

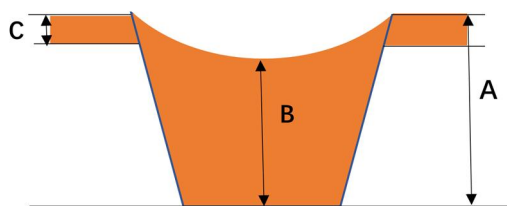


Figure 2. Schematic diagram of the blind hole structure.

2.7. Reliability measurements

As for the tin immersion thermal shock test, the test plates were baked at 150 °C for 6 h, cooled naturally to room temperature in a drying oven, then placed in a lead-free tin oven at 288 °C for six consecutive thermal shocks, each lasting 10 s, and finally observed by metallurgical microscopy for the presence of holes or cracks in the cross-section of the specimen.

3. Results and discussion

3.1. Structural characterization

Figure 3 shows the FTIR spectra of 1,4-butanediol diglycidyl ether (curve 1) and poly (2-methylimidazole co-1,4-butadiene diglycidyl ester) (curve 2). As shown in the results of the IR spectra of the products, the characteristic adsorption peak of the epoxy group (913 cm^{-1}) can't be observed in the raw material, indicating the ring opening reaction of the epoxy group has been carried out in the reactants. In addition, a large number of hydroxyl peaks absorption peaks (3050 cm^{-1} to 3700 cm^{-1}) were observed in the product (curve 2), suggesting that the ring-opening reaction of the epoxy group and a large number of hydroxyl groups proceeded during the synthesis process^[43].

The ^1H NMR hydrogen spectra of the different raw materials and products are shown in **Figure 4**. It can be seen that the position at $\delta = 2.65 \times 10^{-6}$ and 2.85×10^{-6} of 1,4-butanediol diglycidyl ether correspond to the epoxy bond. The resonance of the imidazole ring disappears, and the peak pattern and chemical shift of the hydrogen on the imidazole ring change after the reaction, suggesting the reaction is completed.

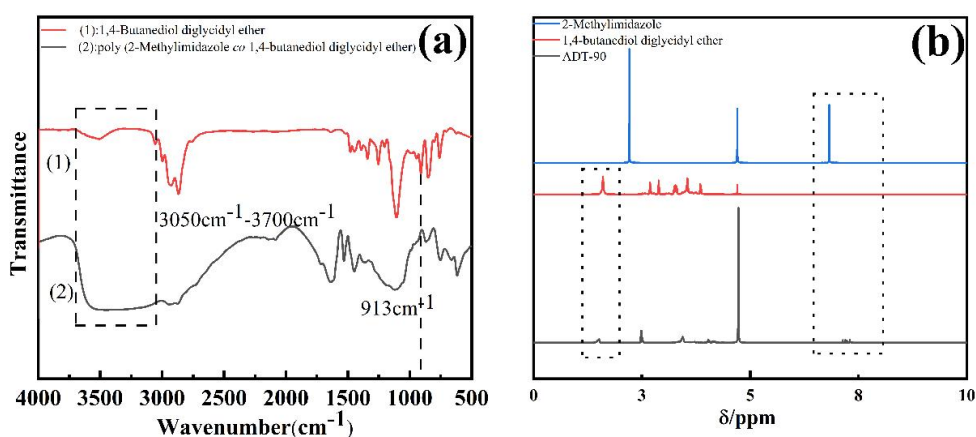


Figure 3. (a) Infrared spectra of 1,4-butanediol diglycidyl ether and poly (2-methylimidazole copolymer 1,4-butanediol diglycidyl ether; and (b) ^1H NMR spectra of two raw materials and ADT-90.

Table 1 shows the heavy average molecular weight (MW) and number average molecular weight of ADT-90. In addition, the MWD value is 1.04, which demonstrates the high purity of the synthesized ADT-90.

Table 1. The GPC result of ADT-90.

Weight-average molecular weight (MW)	Number-average molecular weight (Mn)	Molecular weight distribution (MWD)
1421	1369	1.04

3.2. Analysis of electrochemical behavior during the electroplating process

Typically, the working electrode is set at different speeds to simulate the convection inside and outside the blind hole; in this paper, 1000 rpm and 100 rpm are set to simulate the flow rates inside and outside the blind hole, respectively. (1) and (2) (in **Figure 3**) are the time-potential curves for 2-methylimidazole and 1,4-butanediol diglycidyl ether, respectively. As shown in **Figure 4a,b** PEG and SPS were injected sequentially into the VMS, and polarization and depolarization were observed sequentially, as expected. When 2-methylimidazole and 1,4-butanediol diglycidyl ether were added to the VMS, there was no potential change. This result indicates that neither the reactants 2-methylimidazole nor 1,4-butanediol diglycidyl ether interfered with the plating process.

Furthermore, as shown in **Figure 4c**, the potential difference ($\Delta\eta = \eta_{100} - \eta_{1000}$) obtained from GMs at different rpm can be used to determine whether the additive is effective for microporous fillers according to the convection-dependent adsorption (CDA) mechanism. In general, the value of $\Delta\eta$ is a measure of filling performance, and when $\Delta\eta > 11$ mV, it indicates that the plating liquid system has super-pore filling capability. The results of GMS injected sequentially with PEG, SPS, and ADT-90 at 100 and 1000 rpm, respectively, are shown in **Figure 4c**. The addition of PEG and SPS at different speeds results in negligible potential differences ($\Delta\eta < 0$ or close to 0). However, with the addition of ADT-90, the potential starts to shift sharply negative, with $\Delta\eta$ changing from 0 to as high as 27 mV, implying that ADT-90 is a strong inhibitor of copper ion reduction and can induce significant CDA behavior in the plated liquid system^[18,44].

The chloride ion is an important additive that has a synergistic effect with PEG and SPS. In order to investigate the synergistic relationship between Cl^- and ADT-90, CV tests were carried out on the plated liquid system. **Figure 4d,e** show the results of the CV tests at 100 rpm and 1000 rpm, respectively. **Table 2** shows the integral copper stripping peak area, which corresponds to the amount of copper deposited at the cathode in the CV test. It can be seen that ADT-90 significantly inhibits the deposition of copper when the plating solution is free of chloride ions, and this polarization is more pronounced at 1000 rpm than that at 100 rpm. The addition of chloride ions further enhanced the inhibitory effect of ADT-90, indicating a synergistic effect of ADT-90 and chloride ions at both 1000 rpm and 100 rpm. The inhibition was significantly reduced with the addition of PEG, suggesting that PEG and ADT-90, as well as chloride ions, had a competitive adsorption effect. Finally, the addition of SPS had a slight depolarizing effect at both 1000 rpm and 100 rpm.

As shown in **Table 2**, the amount of copper deposited in the original VMS at 1000 rpm (integral peak area) was greater than 100 rpm. However, after the addition of ADT-90, the situation was reversed. This result indicates that ADT-90 can strongly inhibit copper deposition on the surface of blind holes. The CV results are consistent with those obtained by GM.

Table 2. Integral values for peak copper stripping area in CV tests.

	VMS (without Cl^-)	VMS (without Cl^-) + ADT-90	VMS (without Cl^-) + ADT-90 + Cl^-	VMS (without Cl^-) + ADT-90 + Cl^- + PEG	VMS (without Cl^-) + ADT-90 + Cl^- + PEG + SPS
100 rpm	2.74×10^{-3}	2.55×10^{-3}	1.40×10^{-3}	1.11×10^{-3}	1.03×10^{-3}
1000 rpm	2.82×10^{-3}	2.38×10^{-3}	9.67×10^{-4}	8.70×10^{-4}	6.81×10^{-4}

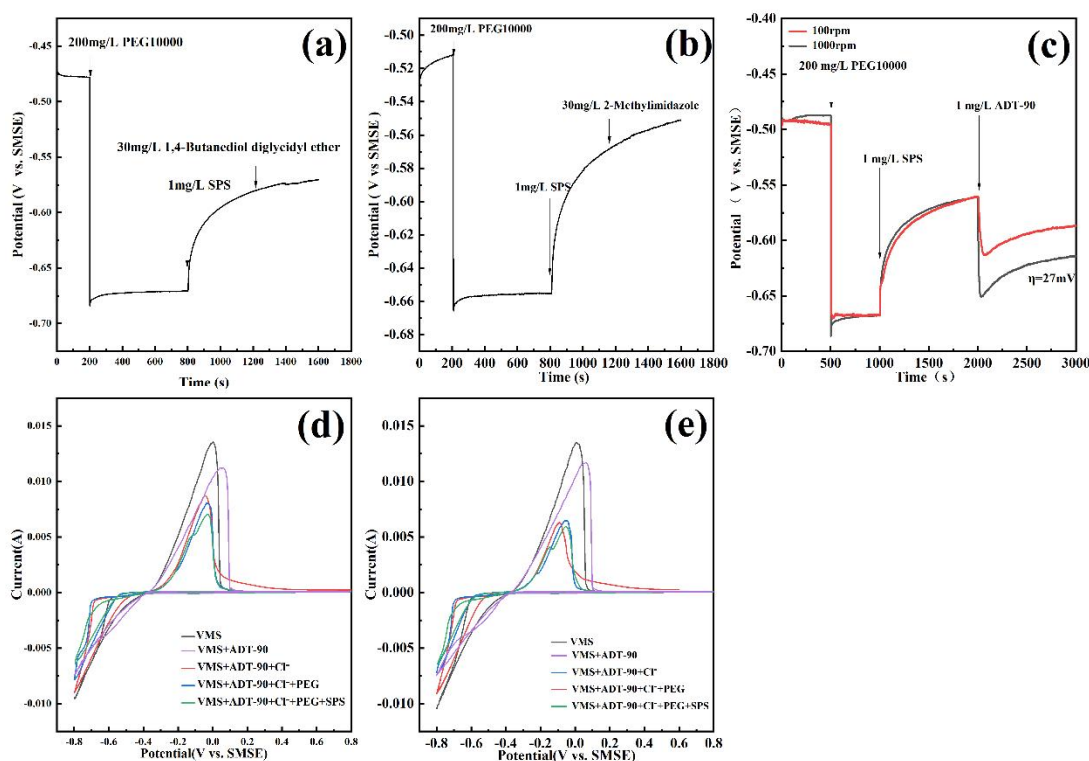


Figure 4. (a) time-potential curves for the addition of 1,4-butanediol diglycidyl ether; and (b) 2-methylimidazole in VMS at 1000 rpm and a current density of 1 A/dm²; (c) constant current measurements with the addition of PEG, SPS, and ADT-90 at different speeds; (d) cyclic voltammetry tests at 100 rpm; and (e) 1000 rpm for different additive plating solutions.

3.3. Blind hole filling test

Due to the strong CDA behavior caused by ADT-90, this molecule has been tested as a leveling agent for pore filling in electroplating. The plating bath formulation consists of 220 g/L CuSO₄·5H₂O, 60 g/L H₂SO₄, 60 mg/L Cl⁻, 400 ppm PEG 10,000, 1 ppm SPS, and 1 ppm ADT-90. After conducting three parallel experiments, the results, as shown in **Figure 5**, indicate an average pore-filling rate of up to 98.3%, which meets the production requirements.



Figure 5. (a), (b), and (c) are cross-sections of the blind holes for each of the three plating experiments.

3.4. Reliability measurements

After the electroplating test, reliability testing was conducted on the plated test boards. Three parallel experiments were performed, and the results are shown in **Figure 6**. After the reliability testing, the test boards showed no signs of blistering or delamination in the plated layer. This indicates that the thermal stress release of the copper plating is well maintained, meeting the production requirements.

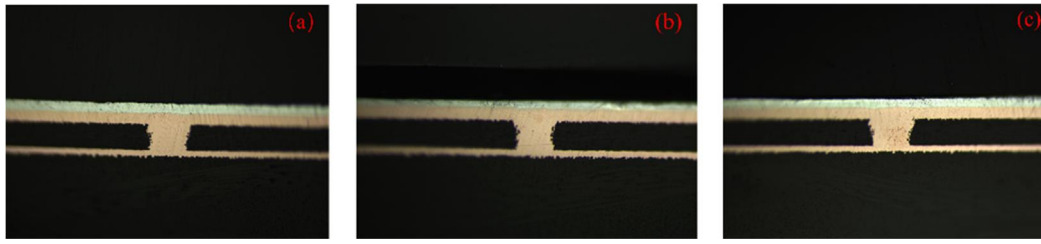


Figure 6. (a), (b), and (c) are metallographic cross-section of a blind hole after thermal shock test.

3.5. Analysis of the corrosion resistance of the coating

After the addition of ADT-90 in the copper plating solution, it demonstrates a significantly improved hole-filling rate during the electroplating process. This enhancement has resulted in the successful passage of the thermal stress test by the test board. However, the corrosion resistance of the copper plating layer is also crucial for PCB boards. Therefore, a preliminary study was conducted to evaluate the corrosion resistance of the copper plating layer.

3.5.1. Electrochemical analysis

Figure 7 shows the Tafel curves for the plated layer with the addition of ADT-90 and the base plating solution. Table 3 displays the corresponding corrosion potential and corrosion current density. It can be observed that after the addition of ADT-90, there is a positive shift in the corrosion potential, indicating an improvement in corrosion resistance. Additionally, the corrosion current density decreases, further indicating improved corrosion resistance. Indeed, it can be concluded that the addition of ADT-90 significantly enhances the corrosion resistance of the plated layer.

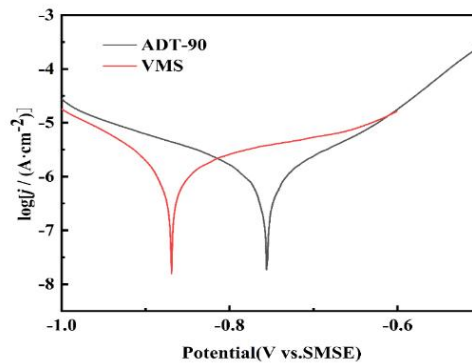


Figure 7. Tafel curves of the ADT-9.0. and VMS.

Table 3. Parameters obtained by fitting Figure 7.

	ϕ_{corr} (vs. SMSE)/V	$j_{\text{corr}}/(\text{A} \cdot \text{cm}^{-2})$
VMS	-0.75	1.296×10^{-6}
ADT-90	-0.86	2.134×10^{-6}

3.5.2. Characterization of phase and structure

As shown in Figure 8a, the addition of additive ADT-90 will alter the crystal orientation of electroplated copper. The peak intensity of the (111) orientation increases significantly, while the peak intensity of the (220) orientation is slightly enhanced. However, the peak intensity of the (200) orientation is slightly weakened. In the previous report, the orientation of (111) crystal planes in electroplated copper enhances its corrosion resistance^[44,45]. Figure 8b is the contact angle of the base plating solution, while Figure 8c represents the contact angle after the addition of ADT-90. The contact angle of the base plating solution is 83.974°, and after

the addition of ADT-90, the contact angle increases to 112.627° . When ADT-90 is added to the plating solution, the copper (111) crystal surface of the plating layer is enhanced, resulting in improved copper corrosion resistance. As a result, the plating layer is not easily wetted by the corrosion solution and the contact angle increases. The addition of ADT-90 enhances the hydrophobicity and corrosion resistance of the coating, which is consistent with the conclusions from the XRD pattern and Tafel tests.

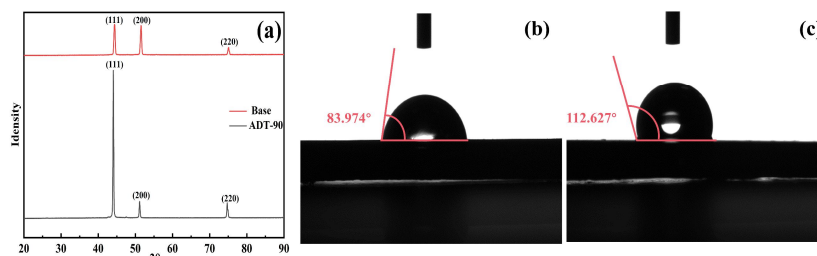


Figure 8. (a) XRD diagram of copper plating with the base solution and plating solution with ADT-90; (b) the contact angle of the base plating solution; and (c) the contact angle after the addition of ADT-90.

4. Conclusion

The synthesis of the nitrogen-containing heterocyclic compound ADT-90 is discussed in this paper. In comparison with other leveling agent related literature, the synthesis and purification methods used in this paper are simple and reduce the cost of the synthesis process. Moreover, this paper uses water as the solvent for synthesis instead of organic solvents, which is more environmentally friendly than other synthetic leveling agent methods. The organic structure of ADT-90 was characterized, and its electrochemical properties were measured. FT-IR test and hydrogen NMR spectra together proved the occurrence of a ring-opening reaction, and the GPC test proved the high purity of the sample. GM showed that neither 2-methylimidazole nor 1, 4-butanediol diglycidyl ether inhibited copper deposition, but the addition of ADT-90 resulted in a potential difference of 27 mV for polarization. CV tests further demonstrate that ADT-90 can strongly inhibit surface copper deposition on PCBS. In addition, XRD shows that the addition of additives can significantly enhance the crystal orientation of copper (111), which is consistent with the results obtained by the contact Angle test. The electroplating verification experiment and plating reliability test showed that the plating copper obtained from the plating solution containing ADT-90 meets the requirements of production. Based on the above research results, the ADT-90 synthesized in this paper is a potential new type of copper plating leveling agent.

Author contributions

Conceptualization, XZ, QZ (Qiuman Zhou), ZP and GH; methodology, XZ and QZ (Qiuman Zhou); software, XZ, ZP, QZ (Qiuman Zhou) and NH; validation, XZ, LH and ZY; formal analysis, XZ and QZ (Qiuman Zhou); investigation, XZ and QZ (Qiuman Zhou); resources, ZP; data curation, XZ and QZ (Qingming Zeng); writing—original draft preparation, XZ and QZ (Qiuman Zhou); writing—review and editing, XZ and QZ (Qiuman Zhou); visualization, XZ and QZ (Qiuman Zhou); supervision, XZ and ZP; project administration, ZP; funding acquisition, ZP. All authors have read and agreed to the published version of the manuscript.

Acknowledgments

This work was supported by Electroless nickel palladium gold plating project applied to PCB (No. GDTP2021A00572), Natural Science Foundation of Guangdong Province, China (No. 2016A030313704), Science and Technology Planning Project of Guangdong Province, China (No. 2016B020240003).

Conflict of interest

The authors declare no conflict of interest.

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