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Research article

The effects of complex agent and sintering temperature on conductive copper complex paste

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ABSTRACT

In this research study, the formulation of precursor-type Cu pastes was done by mixing copper (II) acetate and 2-amino-2-methyl-1-propanol (AMP). Accordingly, the influence of the complex agent amount on the Cu pastes stability was examined at diverse mole ratio ratios. The Cu paste's optimal formulation was then obtained with the copper acetate to AMP ratio of 0.5. The Cu paste with a CuA/AMP ratio at 0.5 was then coated onto glass substrates; they were then sintered under an N_2 environment at various temperatures including 140, 180 and 220 °C. Characterization of the copper complex paste was done by Fourier transform infrared (FTIR) spectroscopy, UV–vis spectroscopy as well as Thermal analysis. Pattern's characterization was also done through X-ray diffractometry (XRD), field emission scanning electron microscope (FE-SEM) as well as four-point probe method for the purpose of confirming the related crystal structure, microstructure and electrical conductivity, respectively. It is demonstrated how the sintering temperature could affect the Cu film. The printed patterns on the glass substrate which was cured at the temperature of 180 °C displayed metalized cooper with low resistivity (30 μΩ cm) and dense copper films.

1. Introduction

Many printing techniques like inkjet, gravure and screen printings have been of much interest recently due to their inexpensiveness and large-scale manufacture for electronic devices, as well as the possibility of being used for being applied to flexible electronics. These printing processes need suitable inks as well as pastes. Conductive pastes have recently found different uses in the printed electronics industry. Examples are fabricating antenna and using wiring on some flexible substrate for ultra-high frequency applications as well as flexible interconnections [1, 2]. While silver paste is known as one of the most widely known ones owing to the silver's considerable conductivity as well as good oxidation resistance, Cu-based pastes can be regarded as an attractive alternative since Cu has some bulk resistivity value which makes it comparable to that of silver (1.68 vs 1.59 μΩ cm, respectively); however, the involved costs are 5–10 times less per ounce and can have more compatibility in comparison to silver with lead-free solders, which could make reflow-based soldering possible [3]. The main problem in regard to copper-based pastes is, however, the rapid rate of oxidation in air, throughout storage as well as sintering processes. To prevent the oxidation of copper in the very process of sintering, some reducing gases like hydrogen or vaporized formic acid have been commonly introduced for suppressing the oxidation occurring throughout the sintering process. Despite all this, the reducing gas is usually dangerous, and atmosphere control needs some specific devices that can make the considered system more complex [4].

The conductive pastes, which include some metal ion complex paste as well as a metal−organic decomposition (MOD) paste, have been examined for the purpose of overcoming the limitations which are related to the nanoparticle-based inks (i.e., ensuring their stability, scalability as well as reduction of costs). MOD pastes belonging to the Cu ion paste are made of copper salts, ligands as well as a number of additives; this ink can be decomposed, forming a copper film that is sintered. For the MOD paste, the gas which is generated during the thermal decomposition may have interference with the formation of a uniform conducting film. Therefore, MOD copper paste can overcome the copper oxidation in sintering [5]. A sample reaction is represented here for copper (II) acetate.

$$Cu(CH_3COO)_2 \rightarrow Cu + 3H_2 + CO_2$$

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Kim et al. provided the demonstration that the film's ultimate electrical resistivity could be dependent on the copper's concentration in some copper (II) formate (CuF)–hexylamine complex which is responsible for controlling the film's porosity as well as impurity content. The least electrical resistivity was achieved from the ink (with the mole ratio of Cu to hexylamine being 1 : 2.6) when annealed at the temperature of 200 °C; this is then followed by the decline of formic acid at the temperature of 250 °C, was 5.2×10^{-8} Ω m [6]. Yabuki et al. also revealed that formed Cu nanoparticles' size depended on the amine type applied in the Cu(II) complex ink [7]. Choi et al. applied complexes of copper formate precursor, diverse hexylamine complex agents (with the mole ratio of Cu to hexylamine being 1: 1 to 1: 4) and Ethyl celluloses as an adhesion promoter or binder. The minimum resistivity that could be achieved was 8 μΩ cm for 1 min in air; subsequently, it was reduced for a period of 5 min under formic acid atmosphere at the temperature of 250 °C [8]. Huang et al. also focused on some thermally triggered self-reducible copper-based MOD ink. They applied CuF to serve as the precursor, monoisopropanol amine to act as the ligand, and octylamine to serve as the co-complexing agent; further, polyvinylpyrrolidone was added to the ink to promote film printability and adhesion on substrate to get the resistivity of 2×10^{-7} Ω m following sintering at 140 °C for 5 min under N_2 environment [9].

To the best of our knowledge, The effect of co-complex agent and sintering temperature on MOD paste with copper acetate (CuA) precursor has not been investigated.

In this research work, therefore, copper acetate was applied as the copper salt for the preparation of the copper complex in the MOD pastes; then the influence of complexing agent to copper salt ratios on the stability of the copper paste was investigated. Subsequently, following the selection of the optimal complexing agent to copper salt ratio, it was printed on a glass substrate and sintering was done in the neutral nitrogen gas atmosphere.

2. Experimental

2.1. Chemicals as well as materials

Copper (II) acetate monohydrate (CuA), 2-amino-2-methyl-1-propanol (AMP), isopropyl alcohol (IA), ethylene glycol (EG) and acetic acid (AA) were got from Merck Chemical Co. Deionized water (DW) was got from ghatran shimi Co (Tehran, Iran).

2.2. Preparation of the Cu ion paste

The real Cu concentrations in the MOD pastes, which were formulated from CuA: AMP = 2:1 (MOD1), 1:1 (MOD2), 1:2 (MOD3) and 1:3 (MOD4), were like the obtained mole-calculated values. A typical procedure for a sample having the molar ratio of CuA to AMP 1:2 is described below.

First, mixing of 3 g of CuA, 2 mL of EG and 3 mL of DW was done by using a conditioning mixer. Then, addition of AMP (2.7 g), 2 mL of AA and 1 mL of IA to the solution was done by gently mixing at the temperature of 50 °C for a period of an hour. Following that, a paste which contained copper-AMP complex ions were got for the purpose of screen printing.

2.3. Preparation of copper films

Glass substrates were cleaned with ethanol; then they were dried by air. The as-prepared MOD paste with a CuA/AMP ratio at 0.5 was then deposited on glass substrates by applying a doctor blade (the applied thickness of the printed areas was 40 μm) and an 85-mesh screen-printing device. For the purpose of thermal sintering, the mentioned films were positioned on a hot plate at diverse temperatures of 140, 180 and 220 °C, under an N_2 environment, at a 5 L/min flow rate, for a period of 10 min.

2.4. Characterizations and measurements

Both thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements were employed in the simultaneous thermal analyzer (DCS/TGA Discovery STD Q600, TA Instruments), at the 10 °C min^{-1} heating rate for patterns, under an argon gas atmosphere. X-ray diffraction (XRD) patterns could be got through applying an X-ray diffractometry (AW/XDM 300, Asenware, China) with a Cu-Kα radiation source (λ = 1.51418 Å). The films' surface morphology was characterized through employing field emission scanning electron microscopy (MIRA3 LMU, TESCAN, Czech Republic). The light absorption properties of the copper complex pastes were then probed by means of the UV spectra recorded on a UV-Vis spectrophotometer (Pishro Pajuhesh Electron Engineering Company, NanodropAr 2015 model, Iran).

The chemical states of the MOD3 complex were analyzed through Fourier transform infrared spectroscopy (Bruker TENSOR 27, Germany). The samples' adhesion strength test was conducted based on ASTM D3359 standard by applying Cross-Cut method; they were utilized to accurately measure the adhesive strength between the MOD paste and the glass. Further, the copper films' volume resistivity was calculated from the surface resistivity by applying a 4-point probe method (Safir Soraya Sepahan Company, FPP100 model, Iran); the film's thickness was assessed through observing a cross-section by applying an optical microscope (OM; Model PMEB, Olympus, Japan).

3. Results and discussion

3.1. CuA–AMP complexes' stability

The evaluation of the solubility as well as stability of the MOD pastes of the Cu ion paste at room temperature, according to the AMP concentration (Figure 1), Figures. 1(c) and (d) show that it was dark-blue at MOD3 and MOD4 (1:2 and 1:3 M ratios of CuA and AMP) and light blue at MOD1 and MOD2 (2:1 and 1:1 M ratios) as shown in Figure 1(a) and (b), owing to the low Cu ion content. This, thus, revealed that the pastes had become darker blue color with the rise of the mole content of AMP, relative to CuA.

Preliminary testing showed that in regard to MOD1 and MOD2, the Cu's ion content was low; this, therefore, made fabrication of some homogeneous film difficult by the ions clustering since they would be subject to drying and sintering processes. The precipitation phenomenon at MOD4 could be confirmed by some Pourbaix diagram. Cu ions formation could be ascribed to the citric acid presence. In this process, combination of them is done with a carboxyl group (COOH) having the most significant solubility degree. So, if there is carboxyl group at a content level which is low, there will be low solubility, thus making the ionization of Cu very difficult. So, precipitation of it is in the form of an oxide or hydroxide [8, 10]. In this article, the color (dark navy color) and

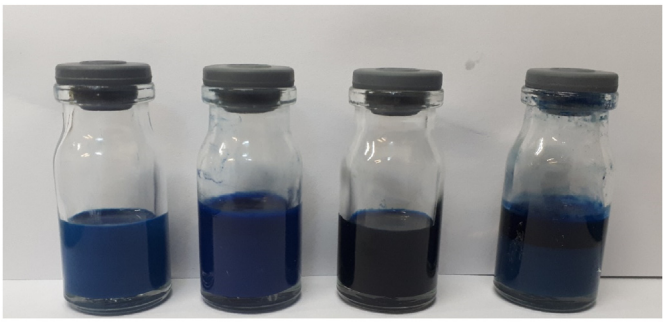

Figure 1. Photographs of different CuA-AMP complexes with various mole ratios of copper acetate versus AMP: (a) MOD1 (2:1 M ratio), (b) MOD2 (1:1 M ratio), (c) MOD3 (1:2 M ratio), and (d) MOD4 (1:3 M ratio).

single phase of the solution were considered as evaluation factors for the formation of a stable and complete complex. So, the MOD paste with MOD3 (CuA/AMP ratio at 1/2) was selected to serve as a suitable paste for further experiments and screen printing.

3.2. Characterization of the MOD complex

FTIR spectrum of the MOD3 complex can be seen in Figure 2. The O–H stretching as well as N–H stretching bands could be observed at 3287 and 3412 cm^{-1} for the considered complex, respectively; they were originally from the AMP ligands in the investigated complex. Further, the characteristic absorption bands in the 2700–3000 cm^{-1}, which corresponded to the considered C–H stretching vibration for $–CH_3$ and $–CH_2$, could be observed. The observed difference in the N–H stretch vibration (3280 and 3400 cm^{-1}) and N–H vending (1340 and 1600 cm^{-1}) bands between CuA and AMP, thus, suggested the amino groups of AMP were coordinated to the Cu surface (II). This also indicated that Cu (II) was protected by AMP ligands [11, 12].

The solution which contained the mentioned complex was subjected to analysis by UV- visible spectrophotometry; its related spectrum can be observed in Figure 3. The relevant absorption band which was at 280 nm could be regarded as a ligand to metal charge-transfer band, which results from an electron transfer from an electron-donating group in AMP to the electron-accepting metal ion. The electrons' lone pair on the secondary amine may also beat work in the formation of the complex. The other absorption band at 715 nm could be ascribed to an electronic d–d transition, which is common for the vast majority of copper complexes [13, 14].

3.3. Thermal decomposition behavior of the MOD complex

The TGA as well as DSC curves related to the MOD3 complex paste under argon are displayed in Figure 4. Figure 4 represents the continuous weight loss, which started at room temperature and ended at ~170 °C. The DSC curve showed some endothermic peak at the temperature of 158 °C (The MOD3 decomposition occurred from 134 °C onward and was complete at ~178 °C), corresponding to the MOD3 thermal decomposition, thus indicating that the paste could be cured at 158 °C. As shown by the TGA results, about 88% of the MOD3 total weight should be vaporized to form a metallic copper film. No further weight loss occurred at annealing temperatures above 170 °C. Thus, the sintering temperatures of 180 and 220 °C could be appropriate for the complete reduction [15].

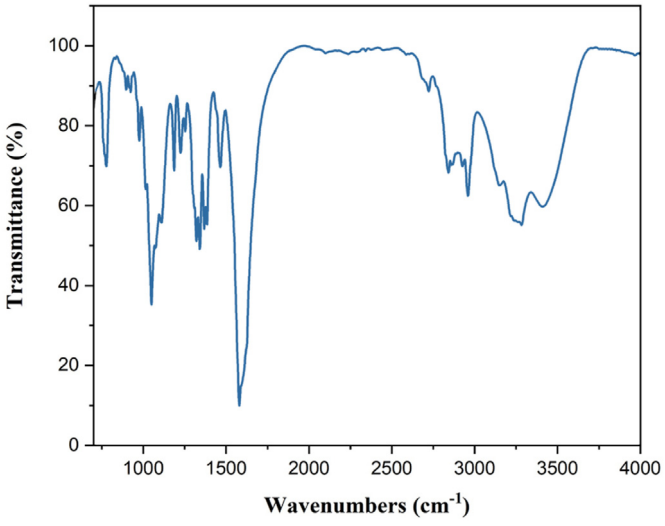

Figure 2. FTIR spectra of the MOD3 complex (molar ratio, CuA: AMP = 1:2).

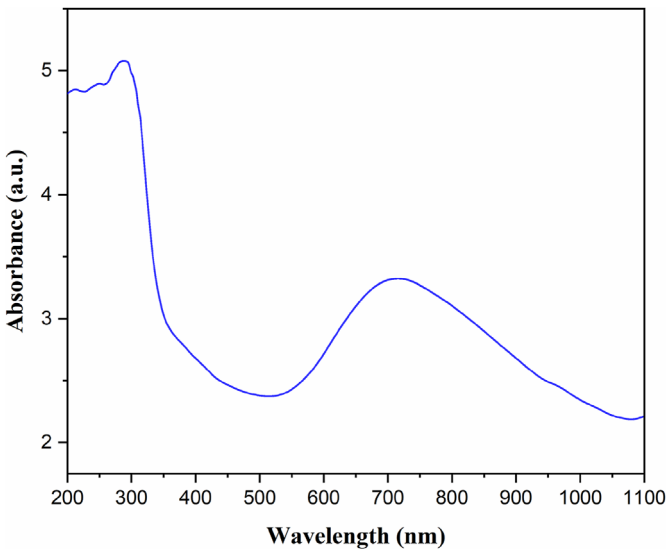

Figure 3. UV–vis spectrum of the MOD3 complex.

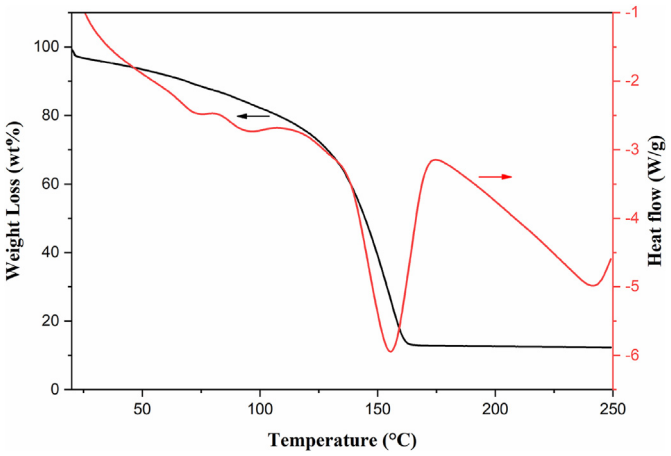

Figure 4. TGA and DSC analysis of the MOD3 complex.

3.4. Phase compositions of the films

To examine the structure as well as composition of the degradation products of MOD3, X-ray diffraction patterns of the complexes which had been processed at 140, 180 and 220 °C were obtained under an N_2 environment for a period of 10 min, as represented in Figure 4. As presented in Figure 5, three Cu peaks at (111) (200), and (220) planes were detected; such peaks could be attributed to the face-centered cubic Cu crystal structure. The impure phases which corresponded to Cu oxide and other materials were not detected, as shown in Figures 5(a) and 4(b). As can be observed from the diffraction patterns, the rise of the sintering temperature of MOD3 to 220 °C (Figure 5(c)) led to the formation of by-products in the form of copper oxides, which strongly hindered the Cu atoms' mutual diffusion between the particles and reduced the percolation paths formation, resulting in the low conductivity of the printed patterns. The oxide peak at CuO (111) could hardly be observed. Despite this, the noisier character of the background curve could be seen [16, 17].

3.5. Impacts of the sintering temperature on the MOD3 paste films' microstructure as well as chemical composition

Examining the surface morphology of conductive ink patterns using a FE-SEM is a suitable option to check the uniformity of the pattern and

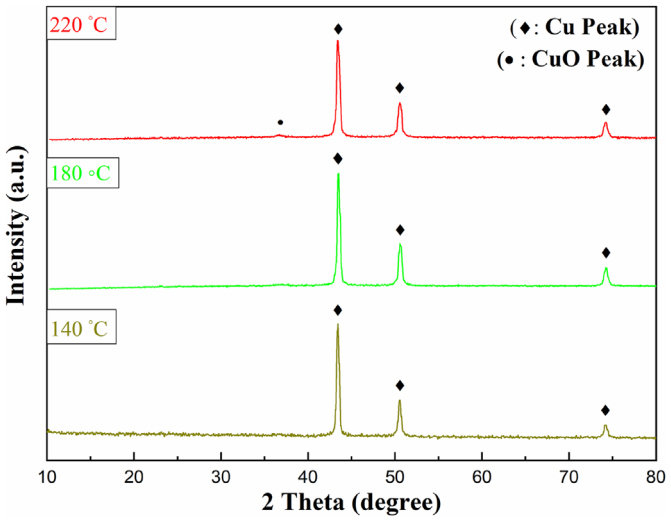

Figure 5. XRD patterns of the MOD3 films which were sintered at the temperature of 140 °C, 180 °C and (d) 220 °C.

interconnectedness of the particles. Therefore, the surface morphology of the Cu patterns which were sintered at different temperatures was observed by FE-SEM (Figure 6). All these films had some microstructure which was made of the interconnected domains belonging to cooper nanoparticles as well as voids.

Such a microstructural feature resulted from the fast evaporation of the solvent as well as the thermal reduction of CuA/AMP complex ions, where many bubbles like H_2 or CO_2 were produced and needed to go through the films, thus led to a structure having pores. The considered film at the temperature of 140 °C (Figure 6a) showed a porous microstructure that consisted of small particles, which were chiefly owing to the observed insufficient decomposition as well as the possible evaporation. The cooper nanoparticles were surrounded or at least covered through organic molecules. With more rise of the sintering temperature to 180 °C (Figure 6b), the microstructure was developed from cooper nanoparticles to the cooper film. The observed grain boundaries became less noticeable. A larger number of particles were in contact at such a temperature too.

When the sintering temperature reached 220 °C (Figure 6c), more cooper nanoparticles were developed. However, some of the pores were observed in the patterns fabricated with the MOD3 paste owing to the fast decomposition of acetic acid and an increase in the proportion of by-products in the form of copper oxides; this could significantly reduce the number of percolation paths, and it had not been fused to form a network throughout the entire copper film. Thus, a quality Cu pattern with low resistivity could be obtained at the temperature of 180 °C [18, 19].

Figure 7 displays the EDS spectrum of MOD3 paste films in the presence of an intense peak of cooper along with other peaks of nitrogen and oxygen. As the temperature of sintering was raised from 140 °C to 180 °C and 220 °C, the Cu content was also increased from 87 (Figure 7a) to 91 (Figure 7b) and 90 wt% (Figure 7c), respectively; meanwhile, the oxygen content was reduced from 6.5 (Figure 7a) to 4.5 (Figure 7b) and 6 wt% (Figure 7c), respectively. In contrast, the nitrogen content was reduced from 6 (Figure 7a) to 4 (Figure 7b) and 3 wt% (Figure 7c), respectively. The results also indicated that the organic molecules were subjected to decomposition and then volatilized chiefly within 140 °C. The observed decomposition or reduction reaction in the case of the mentioned complex was mostly within 180 °C and 220 °C. In addition, in the 220 °C sample, the amount of oxygen was increased, thus indicating that the nanoparticles were oxidized. Furthermore, EDS provided the confirmation that the nanoparticles which were shown in the FE-SEM images were Cu [20, 21].

3.6. Adhesion and resistance of films

Adhesion test samples were evaluated on a scale of 0B (weakest) to 5B (strongest). For the Cu films sintered at 140 °C, 180 °C and 220 °C, 45%, 15% and 25%, they were detached by the tape test and categorized as 1B, 3B and 2B class, respectively. Pores as well as voids' presence in the considered film could weaken the adhesion strength. Many other factors could have an effect on adhesion, such as the surface energy and polarities of the joined surfaces, substrate surface roughness, the solvent residues in the inks or the solvents' absorption into the substrate surfaces [21]. The more detailed researches are needed to well explain the considered adhesion mechanism.

Figure 8 displays the electrical resistance related to the Cu films which had been prepared from the MOD3 complex paste that was sintered at 140 °C, 180 °C and 220 °C for a period of 10 min under an N_2 gas flow.

The obtained volume resistivity in the case of MOD3 paste was found to be 65, 30 and 40 μΩ cm for the purpose of heat treatment at 140, 180 and 220 °C, respectively. The Cu films' volume resistivity was decreased in a range from 140 to 180 °C, which was in agreement with the CuA/AMP complex thermal decomposition temperature (as shown in Figure 4). Despite this, an increase of temperature to above 180 °C was accompanied by an increase in the resistance of the formed layers, which was owing to the increased proportion of by-products in the form of

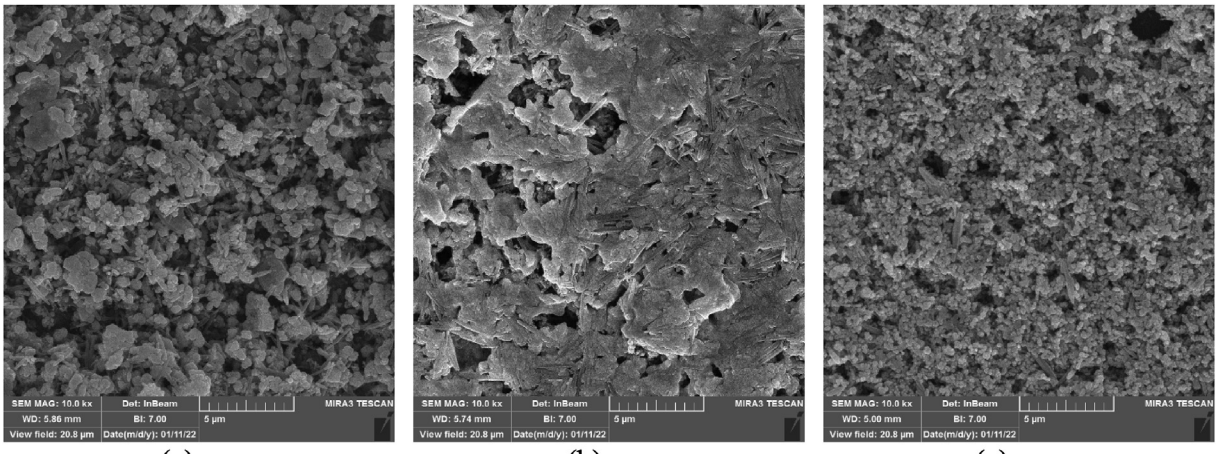

Figure 6. FE-SEM of the copper films of the MOD3 complex which was sintered at: (a) 140 °C, (b) 180 °C and (c) 220 °C.

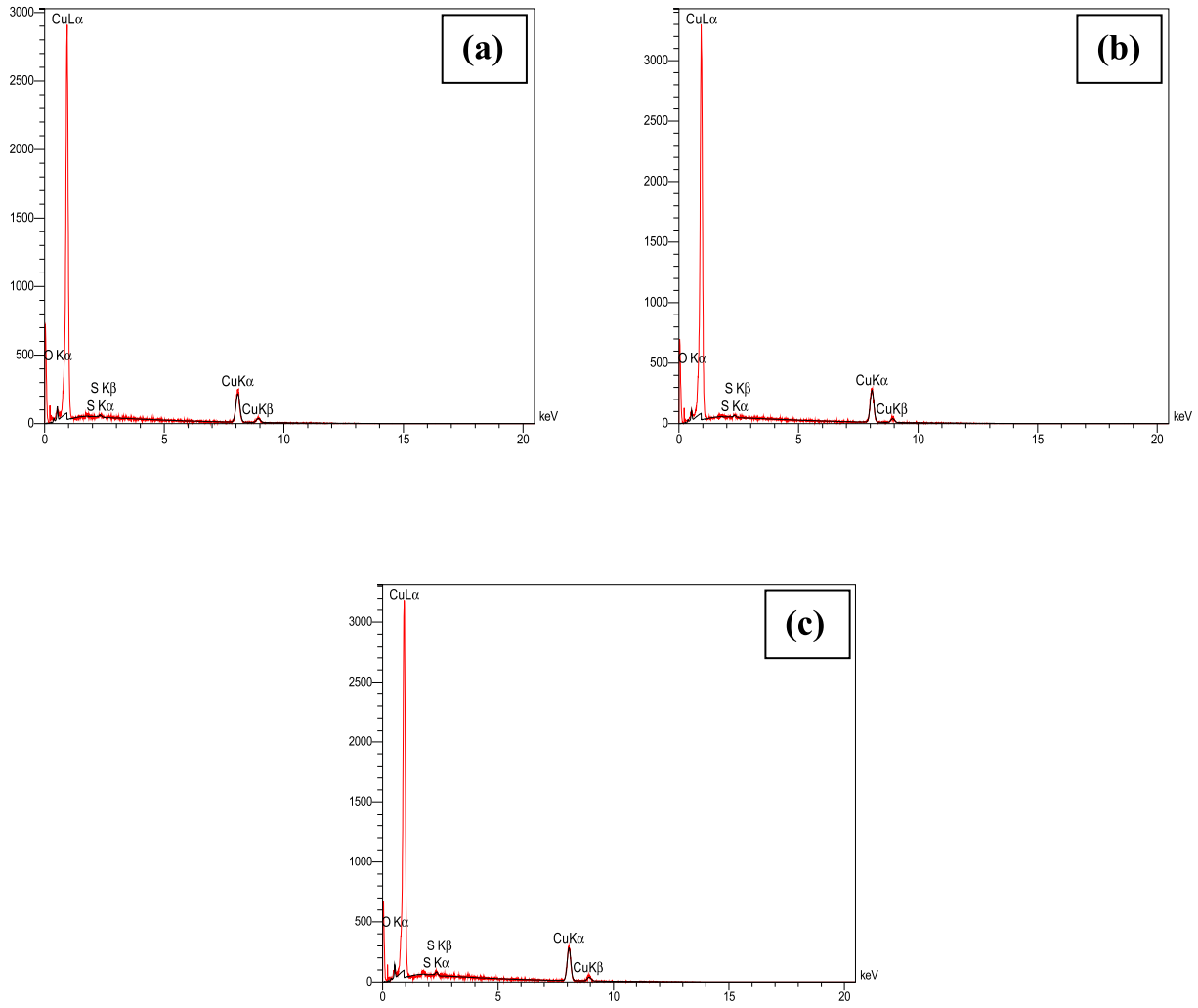

Figure 7. EDS results in the films as a function of the sintering temperature at: (a) 140 °C, (b) 180 °C and (c) 220 °C.

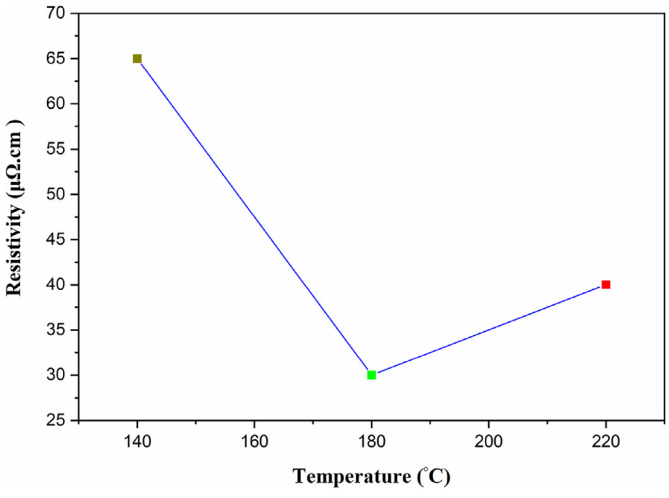

Figure 8. The volume resistivity of the film which was obtained after heating MOD3 paste for a period of 10 min under a nitrogen atmosphere as a function of the heating temperature.

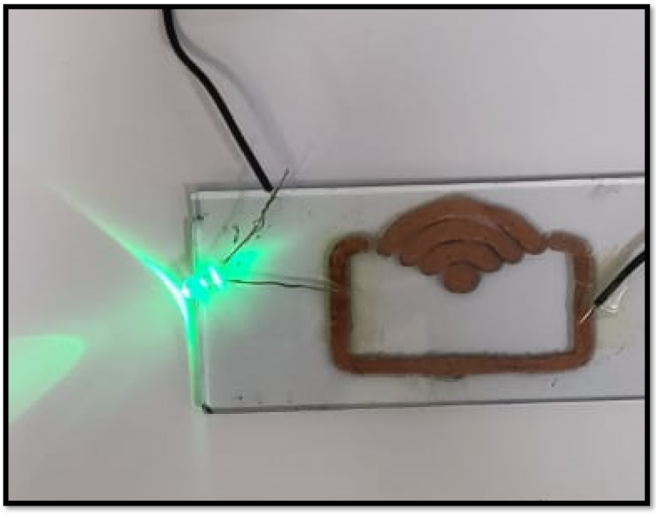

Figure 9. Pictures of a screen-printing pattern which was printed with the optimal MOD 3 paste that was sintered at the temperature of 180 °C on a glass substrate.

copper oxides; this remarkably reduced the number of percolation paths, thus leading to the printed patterns' low conductivity [22]. To show that the MOD3 paste which was sintered at the temperature of 180 °C was appropriate for the purpose of screen printing, it was applied for the fabrication of a pattern for conductive tracks with diverse widths that could be noticed on a glass substrate (Figure 9).

4. Conclusions

In the present research study, pastes were formulated through the control of the mole ratio between copper (II) acetate and AMP for Cu screen printing. The Cu ion complex with the CuA/AMP mole ratio of 0.5 was found to be stable in a solution and there was the generation of no precipitates. Through analyzing the thermal decomposition behavior, it was confirmed that all materials, with the exception of Cu, were completely decomposed at temperatures which were less than 170 °C. The MOD3 paste (CuA/AMP mole ratio at 1/2) was screen-printed onto glass substrates and then sintered at 140, 180 and 220 °C. The obtained pattern's crystal structure was confirmed to be a face-centered cubic one at all sintering temperatures which were considered. Further, the microstructure and electrical resistance were affected by the sintering temperature and the patterns which were formed at the temperature of 180 °C had a well-sintered microstructure and low resistivity, and the highest adhesion level (3B), as compared with those which were created at the temperatures of 140 and 220 °C. When the patterns were created by applying the MOD3 paste that was heat-treated at 180 °C for 10 min in the N_2 atmosphere, the volume resistivity was 30 μΩ cm. Finally, the screen printing of conductive lines was demonstrated by applying the Cu ion complex paste.

Declarations

Author contribution statement

Hamed Naderi-Samani: Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Reza Shoja Razavi, Reza Mozaffarinia: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

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Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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