



TECHNICAL ARTICLE

The Interfacial Phenomena Between Graphene on Cu Substrate Covered by Ni, Cu, or W Layer, with Liquid Ga-Sn-Zn Alloy

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To obtain better parameters of wetting and interfacial properties, the Cu substrate was covered by graphene. The first experiment showed that the liquid metal mechanically destroys the graphene layer. Therefore, the Ni, Cu, or W layer was sputtered to secure the graphene. The obtained graphene was examined by micro-Raman spectrometry. The thickness of the Cu, Ni, or W layer was ~ 25 nm, compared to previous work, where the thickness of the Ni-W electrochemically deposited layer varied from 8–10 μm . To observe changes at the interface, the experiments were performed with Ga-Sn-Zn eutectic alloy using the sessile drop method at temperatures of 100, 150, and 250 °C long-time contact of 1, 10, or 30 days. Atomic force microscopy was used to show the topology of obtained samples. The microstructure observation of the cross-sectioned samples was made by scanning electron microscopy combined with energy dispersive x-ray spectroscopy. The x-ray diffraction was conducted to identify occurring phases at the interface from the Cu-Ga system. The investigation showed that such a very thin Ni, Cu, or W layer is not sufficient to protect the Cu substrate from a reaction with liquid metals such as eutectic Ga-Sn-Zn, which can be used in cooling systems. The performed XRD analysis and microstructure observations show the occurring CuGa_2 phase at the interface and dissolution of the Cu substrate in molten alloy with increasing temperature and time. The interfacial CuGa_2 phase grows very slowly at annealing temperatures below 150 °C for the graphene/Ni and graphene/W coatings. Therefore, these coatings can be used to protect a copper substrate in cooling electronic devices.

Keywords graphene, interfaces, liquid metals, wetting

1. Introduction

The development of electronic connections based on Cu substrate covered by a graphene layer still leaves room for improvement in order secure joints with desired and optimized properties. Typically the soldering process is accompanied by the formation of detrimental intermetallic phases (IMCs) at the

interface of solder and substrate (Ref 1-3). These phases are brittle, therefore they reduce the mechanical strength of the joint and decrease electrical properties. Thus, a large increase in the IMCs layer at the interface between the liquid solder and the substrate usually degrades the properties and reliability of the solder joint. It is, therefore, important to control the growth kinetics of these layers (Ref 2). Therefore, materials and technologies are sought that could prevent the formation of an intermetallic layer or inhibit their growth. In this case, as proposed in the previous paper (Ref 4), the graphene layers were made to protect the Cu substrate in the soldering process using Sn-Zn solder. However, the wetting process and reaction phenomena at the interface are more complex than wetting of the graphene layer by the solder, and comes with the destruction of this layer. Moreover, the aspect of oxidation of graphene is also important which have effect on wetting test (Ref 5). Furthermore, as shown Authors (Ref 6) in the case of wetting SiC sputtered graphene layer by liquid Au, the oxidation and graphene layer quantity have high impact for measured contact angle.

Another big issue is the application of liquid metals in electronic devices. The problem, which must be resolved is the dissolving of Cu substrate by liquid gallium, as shown in (Ref 7-9). In this system the following IMCs phases appear, CuGa_2 and Cu_9Ga_4 . These grow over time, and the growth rate depends on temperature. In a previous study, the interaction between Cu (Ref 8) and Ni (Ref 10) substrates with liquid Ga alloys was investigated. The experiment was carried out for temperatures of 100, 150, and 250 °C and times of 1, 10, and

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30 days. In the case of the Cu substrate, the obtained microstructures of the cross-sections confirm the formation of IMCs phases from the Ga-Cu system, such as CuGa_2 and Cu_9Ga_4 . Thermodynamic calculations confirmed that the Cu_9Ga_4 phase is characterized by the lowest Gibbs free energy, and hence dominates and grows the most with time and temperature. At the highest temperature of 250 °C, it reacts completely with the substrate. The dissolving of the Ni substrate by liquid metal (Ref 10) was slower compared to the Cu substrate (Ref 8), but IMCs of Ga_3Ni_2 and Ga_7Ni_3 layers formed at the interface. When comparing activation energy for these systems it is almost two times higher for Ni (32.3 (kJ/mol) than for Cu (16.9 (kJ/mol)), which is similar to 23.8 (kJ/mol) for pure Ga on Cu (Ref 11). The next step in protecting the Ni substrate was to cover it with a W-Ni layer, which successfully protects the Ni substrate up to 150 °C (Ref 12). To protect the Cu substrate, a graphene layer covered by a thin layer made up of Cu, Ni, or W was proposed.

In this study, the idea was to use a graphene layer to protect Cu in advanced liquid metal based on Ga, which could be used as a coolant in electronic devices (Ref 13) and especially for liquid metal batteries at room temperature (Ref 14). However, graphene also needs to be protected against mechanical wear by liquid metal. In this case, a magnetron sputtered Cu, Ni, or W thin layer was applied. To check the solution, wetting testing was carried out for specific temperatures and times. The received cross-section was observed and analysed using scanning electron microscopy combined with energy dispersive spectroscopy.

2. Experimental

For the Cu 99.99% plate with 250 μm thickness, the sessile drop method was used with a droplet of liquid metal from eutectic Ga-Sn-Zn (90.15 of Ga, 6.64 of Sn and 3.21 of Zn (at.%) corresponding to 86.3, 10.8 and 2.9 (wt.%), respectively, (Ref 15). A graphene coating was applied to the copper substrate by chemical vapor deposition (CVD) (Ref 4). The obtained graphene coatings were tested by micro-Raman spectrometry. Chemical vapor deposition (CVD) was used to cover the copper substrates with a graphene layer. Next, a copper, nickel, or tungsten layer were deposited on the graphene by magnetron sputtering. The μ -Raman characterization of the graphene layer was conducted using an inVia Renishaw Raman spectrometer with an optical Leica microscope (lens 100 \times) in backscattering geometry. The HeNe laser excitation (633 nm wavelength and power of approx. 14 mW) was used. The μ -Raman spectra were collected from areas about 1 μm^2 in three ranges of Raman shifts: the range of bands mainly of metal oxides (50-1000 cm^{-1}), the range of bands D and G (1000 to 1700 cm^{-1}), which are associated with the presence of sp^2 hybridized carbon, and the range of the 2D band for carbon sp^2 (2600-3250 cm^{-1}). The weak Raman bands were visible only in the D/G range (1000 to 1700 cm^{-1}), indicating the presence of a defective graphene layer. For the amplification of the signal to the background in the graphene spectrum, 500 accumulations were used. The thin films of Cu, Ni, or W were deposited onto a graphene layer by a direct current (DC) magnetron sputtering system (EM ACE600, Leica, Germany) from a pure metallic target. The purity of the Cu target was 99.999%, the Ni target was 99.99%, and the W

target was 99.99% (Kurt J. Lesker Company). As a working gas, Ar (99.999%) was used with working pressure between 2×10^{-2} and 8×10^{-3} mbar. No external heating was applied during the growth stage, and depositions were carried out at room temperature. In this study, constant rotation was maintained to make the deposition as homogeneous as possible. The sputtering current was fixed at 60 mA for Cu, 100 mA for Ni, and 90 mA for W. The thickness of obtained films was monitored with a quartz crystal oscillator. The film thickness is 25.4 nm for Cu, 25.5 nm for Ni, and 25.8/50.6/102.2 nm for W. The sessile drop tests were performed at temperatures of 100, 150, and 250 °C and times of 1, 10, and 30 days. The microstructure characterizations of the cross-sectioned samples after the wetting tests were performed using an FEI E-SEM XL30 scanning electron microscope (SEM) equipped with an EDAX GEMINI 4000 energy dispersive spectrometer (EDS).

3. Results and Discussion

To protect the copper substrate, a graphene layer was proposed to avoid the reaction of the liquid eutectic Ga-Sn-Zn alloy with the substrate. Unfortunately, the graphene layer turned out to be insufficient to protect the Cu substrate. It is likely that, at high temperatures, the liquid gallium alloys mechanically destroy the graphene layer. The reaction at the interface between liquid metal and Cu substrate is observed as shown in Fig. 1(a). A similar observation was made in the previous paper (Ref 4)—the graphene coating consists of flakes separated by a boundary of overlapping flakes, which are the weakest link in the graphene layer. The mobility of the flux at high temperatures caused the formation of gaps between the graphene sheets, enabling the reactions of liquid solder with Cu and diffusion processes. As a result, a layer of IMCs phases was formed at the interface and the graphene sheets detached from the Cu substrate and migrated to the top of the solder (Ref 4). To protect the graphene layer, a magnetron sputtered Cu, Ni, or W layer was proposed. The microstructure of what? after the wetting test at 100 °C for 1 h is shown in Fig. 1(b) and (c), where the interface shows a slight amount of reaction product formed due to the interaction of the eutectic Ga-Sn-Zn alloy with Cu sputtered on graphene. For the Ni layer (Fig. 1c), there was no reactively formed interfacial phase. The sputtered Cu or Ni layer could react with eutectic Ga-Sn-Zn alloy as shown in the previous study (Ref 8, 10). However, such a reaction, means formation of IMCs layer at the interface should help to protect the graphene layer from mechanical wear from liquid eutectic Ga-Sn-Zn. The applied W layer, which is not supposed to react with Ga alloys, should help to protect graphene on the Cu substrate, and, as shown in Fig. 1(d), there is no reaction product, similar to Ni cover. Magnetron sputtered Cu or Ni, which react with Ga-based alloy, will form an IMCs layer at the interface that should block the diffusion of Ga to Cu and avoid the resulting dissolve of the Cu substrate. A magnetron sputtered W layer should not, in regard to the Ga-W phase diagram, lead to an interfacial reaction. In all cases, the sputtering of Cu, Ni, or W caused the changing surface appearance and covered the graphene layer on the Cu substrate. However, access to air after the all process allows oxides to form at the obtained surface, as Cu_2O , NiO and WO_3 were detected in Raman spectra, respectively.

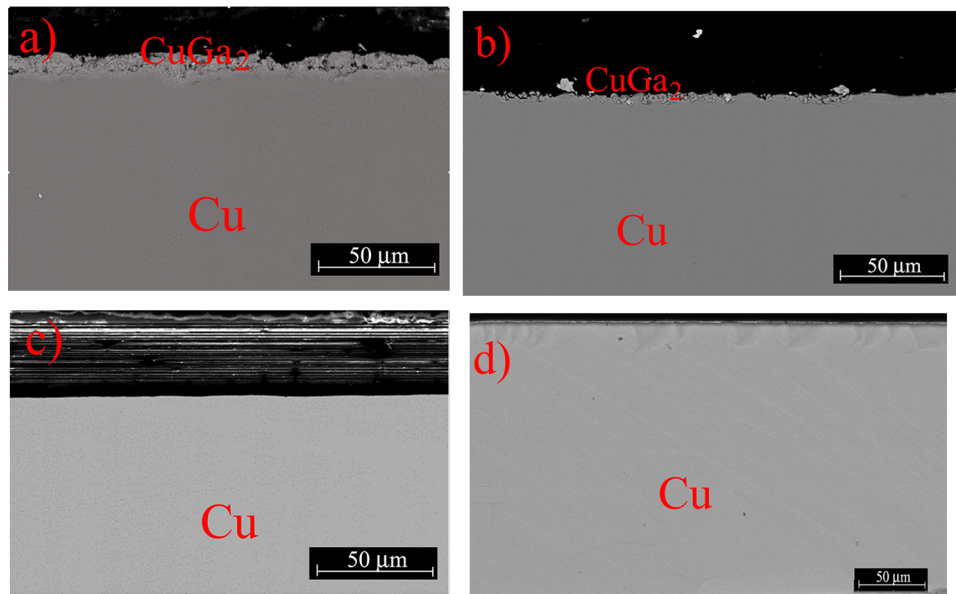


Fig. 1 SEM images showing the microstructure of cross-sectioned sessile drop couples after testing at temperature of 100 °C for 1 day with eutectic Ga-Sn-Zn alloy on Cu substrate: (a) covered by graphene layer, (b) graphene layer covered by Cu, (c) graphene layer covered by Ni, (d) graphene layer covered by W

As shown in (Ref 16), the obtained graphene by CVD method contains defects such as wrinkles, grain boundaries and foldings, which are the line with our previous study (Ref 4), that reason we decide to protect graphene by sputtering metal layer. The obtained samples after the sputtering process, where the tint of the surfaces of the investigated samples changed, giving direct information about the protective layer's presence at the interface. Figure 2, 3, and 4 show the microstructure and Raman's spectra for magnetron sputtered Cu, Ni, and W layers, respectively. Such a thin layer (25 nm) of metal was transparent to both visible light and Raman. The Raman analysis shows that the partially deposited metallic coating is oxidized. In the lower range, NiO is visible for samples with nickel, and Cu₂O for samples with copper (Fig. 2 and 3). However, nickel oxide may still have bands in the range of 1000-2000 cm⁻¹ that may overlap with graphene. In the case of the W protecting layer, the WO₃ was detected. Moreover, in the sputtering process, the graphene coating was severely damaged. The D and G peaks are visible in the range around 1330 cm⁻¹ and 1590 cm⁻¹, and the 2D band is also visible sometimes (Fig. 2-4). Obviously, the strong deterioration of graphene is partly due to the weakening of the detection by the metallic coating.

The microstructure of cross-sections of Cu/graphene with Cu, Ni, and W, after sessile drop tests at 100 °C and 720 h, are presented in Fig. 5, 6, and 7, respectively. Regarding the number of samples for temperatures of 100, 150, and 250 °C and times of 1, 10, and 30 days for all systems (Cu/graphene/Cu, Cu/graphene/Ni, and Cu/graphene/W), the microstructures of the cross-sections are available in supplementary file S1-S9. In the microstructures presented in Fig. 5-7, the EDS line scan is marked. This shows the distribution of chemical elements in the interfacial area. Such changes show the dissolution process of Cu substrate in the liquid alloy, and the formation of IMC at the interface, which was confirmed by XRDs.

The phase identification using XRD shows the appearance of the CuGa₂ phase at the interface (Fig. 8), which is in line with EDS data received during SEM observations. After the

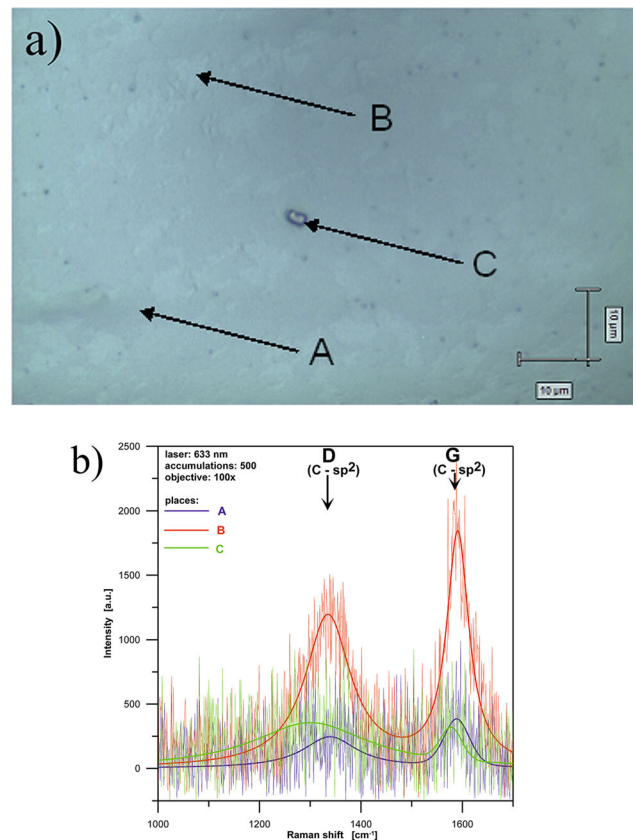


Fig. 2 (a) Microstructure of the layers obtained from the optical microscopy of the Cu/graphene/Cu, (b) Raman spectra: blue line (in BW lowest peaks)—light places (B); green line—dark places (A); red line (in BW highest peaks)—dark spot (C) (Color figure online)

wetting test at 100 °C and 720 h, a small amount of the CuGa₂ phase is observed at the interface. With increasing temperature

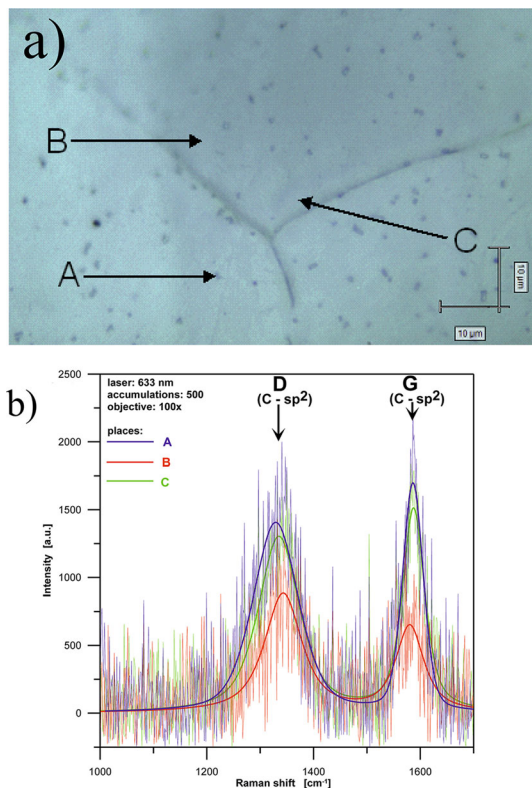


Fig. 3 (a) Microstructure of the layers obtained from the optical microscopy of the Cu/graphene/Ni, (b) Raman spectra: blue line (in BW highest peaks)—dark spot (A); red line (in BW lowest peaks)—dark places (B); green line—light places (C) (Color figure online)

to 150 °C, the amount of CuGa₂ phase at the interface increases, and for 250 °C, the complete dissolution of the Cu substrate is observed. The increasing temperature caused the increasing diffusion of Cu to liquid eutectic Ga-Sn-Zn, which has a key impact on the dissolution of the Cu substrate. As shown in (Ref 17), the driving force in this process is the metallic bond at Ga/CuGa₂ interfaces, which have been demonstrated to be the major contributing factor of the adsorption energy and spreading—wetting phenomenon. The dissolution mechanism is similar to that observed in the literature (Ref 7, 8), dominating the volume diffusion and, at higher temperatures, with a fast pace of dissolution as a reaction at the grain boundaries (high energy places). The formation of IMCs with CuGa₂ or Ga-Ni phases at the interface for Cu/graphene/Cu and Cu/graphene/Ni, respectively, were expected. However, the thickness of the layers and the high diffusion of Cu to liquid caused the dissolution of the Cu substrate despite the graphene layer. The diffusion of Cu could be possible due to line defect as boundaries of flakes and destroy of graphene layer, as observed in previous work (Ref 4). This means that the Cu or Ni layer was not thick enough to create a barrier for liquid Ga and overall to protect the graphene layer.

In the case of the W protective layer, it was assumed from the Ga-W phase diagram that there would be no its reaction with the liquid Ga-based alloy. The continuous W layer after the sputtering process was expected. However, the applied W layer shows a different character with a grain boundaries, and its higher thickness (up to 50 or 100 nm) does not eliminate this phenomenon. Figure 9 shows the observation using AFM of

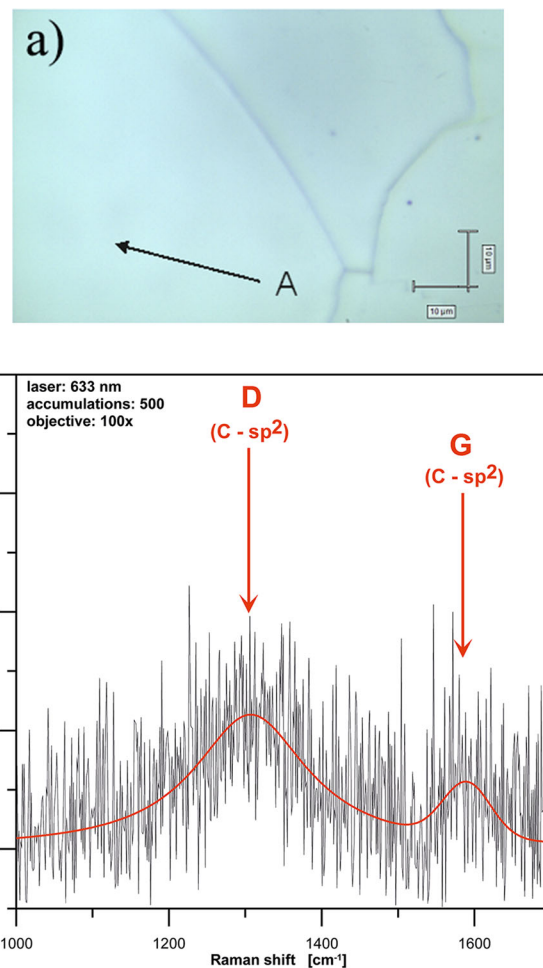


Fig. 4 (a) Microstructure of the layers obtained from the optical microscopy of the Cu/graphene/W, (b) Raman spectra corresponded to marked placed A in microstructure

the Cu/graphene/W sample with a thickness 50 nm of the W layer. As can be seen, there are grains with a different character of growth (such phenomena will be studied in another paper, where sputtering of a W layer on a monocrystalline Cu with graphene will be investigated), with a boundary between W grains. As shown in Fig. 9, the grain boundary reaches the thickness of the applied W layer. It is likely that this is where liquid metal penetrates the boundary to the graphene and, as a result of circulation of Marangoni movements (Ref 18, 19), destroys the protective layer. Taking into account such an effect, until we better understand the sputtering of W layer on polycrystalline Cu, the applied 25, 50, or 100 nm W layer on Cu/graphene cannot be assumed to prevent the Cu substrate from reaction with eutectic Ga-Sn-Zn. During wetting in (Ref 6), shown how important is quantity of graphene—substrate connection, and how big influence have for observed contact angle. Moreover, all defect detected on surface have significant effect on interaction phenomena at interface. In the performed experiment for samples Cu/graphene/Cu, Cu/graphene/Ni, and Cu/graphene/W after interaction test at a temperature of 150 °C and 30 days were compared, and the lowest dissolution of Cu substrate was observed for Cu/graphene/W. At a higher temperature of 250 °C, the process of dissolution of Cu substrate is similar as in the previous study (Ref 4, 8). The

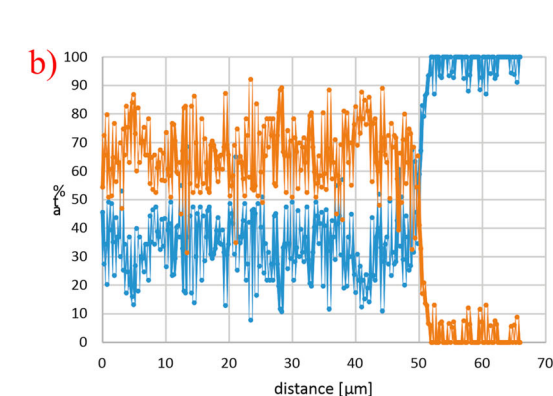
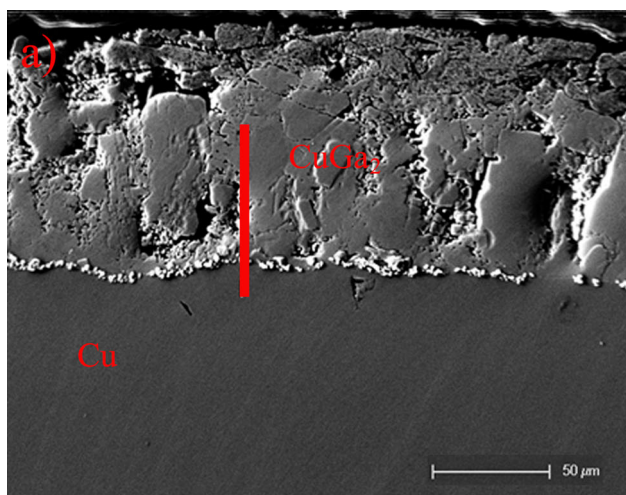


Fig. 5 The cross-section microstructure (a) and line scan EDS (b) in SEM, after wetting of Ga-Sn-Zn alloy on the Cu/graphene/Cu at temperature 100 °C and time 720 h

CuGa₂ phase is created at the interface, and this grows very quickly, consuming the Cu substrate completely after 30 days.

The problem of continuity of the protective layer must be resolved, but the issue of the adhesion of the sputtered layer is also crucial. The proposed electrodeposited layer, as in the previous study (Ref 12) to protect the Ni substrate, cannot be applied in this case because, during the electrodeposition process of Cu, Ni, or W, the graphene layer on the Cu substrate is destroyed. Moreover, the oxidation of graphene after the process or liquid metal has a significant effect on properties (Ref 20, 21), which means such materials require further research to expand their application possibilities. The new trends, as graphene field effect transistors with liquid metal interconnects (Ref 22), where Authors demonstrated transport characteristics for a single-gate graphene field-effect transistor, in addition, the manipulation of the physical characteristics of Galinstan is a precursor to flexible devices, what show how huge have possibility of application graphene with liquid metals connections.

4. Conclusions

The proposed thin Cu, Ni, or W layer to protect graphene on the Cu substrate from dissolution by eutectic Ga-Sn-Zn was investigated. In all cases, the CuGa₂ phase was formed at the

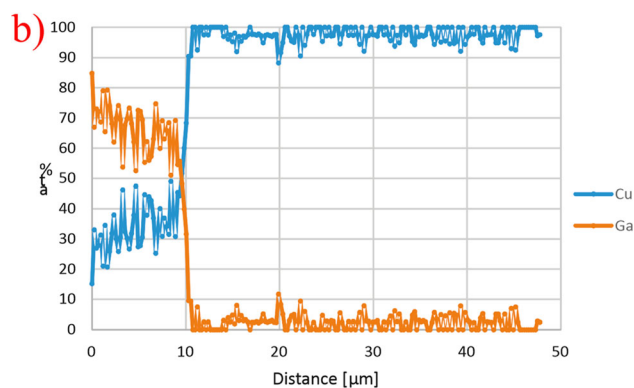
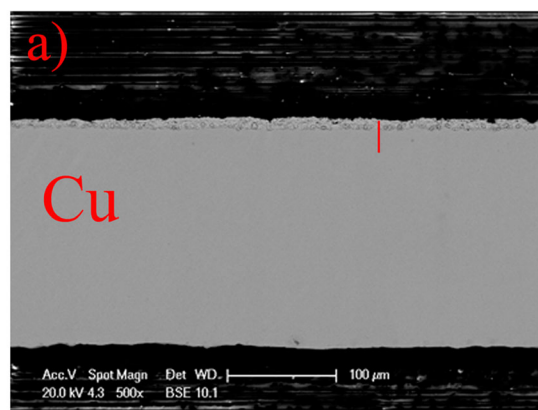


Fig. 6 The cross-section microstructure (a) and line scan EDS (b) in SEM, after wetting of Ga-Sn-Zn alloy on the Cu/graphene/Ni at temperature 100 °C and time 720 h

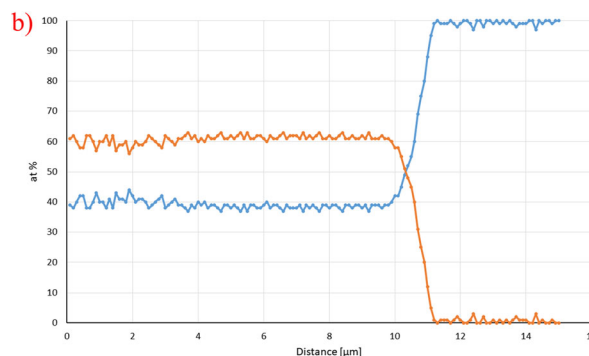
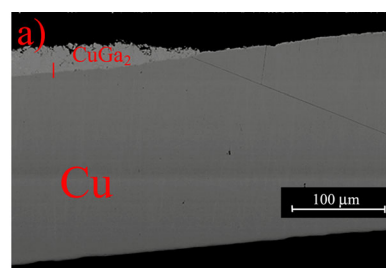


Fig. 7 The cross-section microstructure (a) and line scan EDS (b) in SEM, after wetting of Ga-Sn-Zn alloy on the Cu/graphene/W at temperature 100 °C and time 720 h

interface during sessile drop tests. The CuGa₂ phase was identified by EDS/SEM analysis and confirmed by XRDs. For

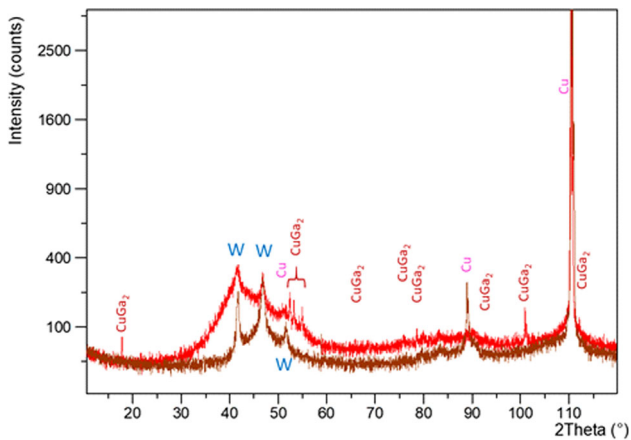


Fig. 8 XRD after wetting of Ga-Sn-Zn alloy on the Cu/graphene/W at temperature 100 °C for 240 h

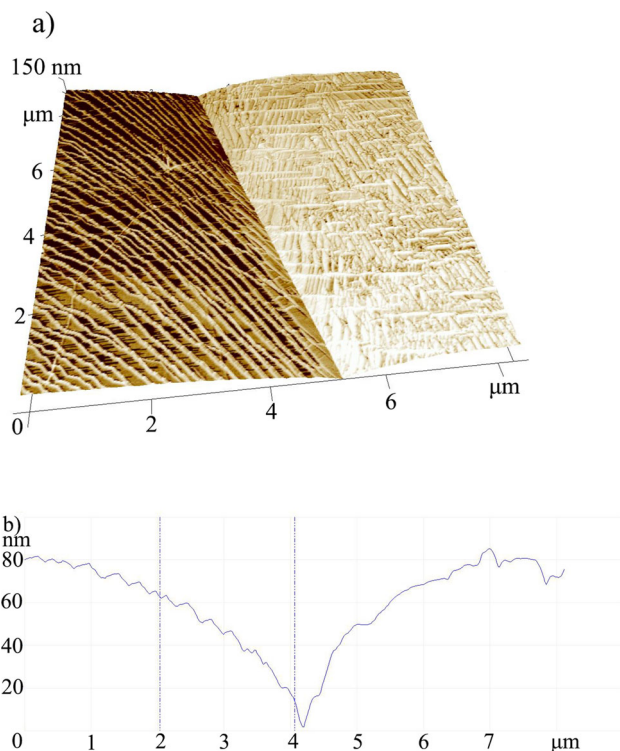


Fig. 9 The AFM observation of the Cu/graphene/W sample with the thickness of 50 nm of W layer

150 °C, the W protective layers show the lowest dissolution of Cu substrate covered by graphene, compared to the Cu or Ni layer. However, if the W sputtering layer is not leak proof, it will not protect the Cu substrate sufficiently. The mechanism of sputtering a thin layer onto graphene needs further study. In the case of the Cu and Ni layers, the created IMC layer at the interface is too thin to protect graphene in the long-term contact with molten alloy used in this study. On the other hand, the SEM observation shows that the CuGa₂ phase grows very slowly at the interface at annealing temperatures below 150 °C for the graphene/Ni and graphene/W coating. Therefore, these coatings can be applied to protect a Cu substrate for cooling electronic devices. Moreover, the proposed protection layer

could be applied for soldering electronic components, where the time of heat treatment is quite short 1 to 3 min.

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