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Study of Reactive Diffusion in Cu/Zn Diffusion Couple

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Abstract

Objective: To study the micro structure of the interfacial region of Cu/Zn diffusion couple at 250°C and its evolution with diffusion time. **Methods:** The couple was prepared by a diffusion bonding technique and then annealed at 250 °C in Argon atmosphere. The micro structure resulting from diffusion reactions in the couple was studied by scanning electron microscopy and energy dispersive X-ray. **Findings:** Two continuous inter metallic phase layers formed at the interface between Cu and Zn metals. The formed inter metallic were ε and γ phases of the Cu-Zn system. The γ layer grew much faster than the ε layer. The growth kinetics of the γ phase layer was parabolic with a growth constant $k_p = (1,9 \pm 0,02)10^{-13} m^2 s^{-1}$. **Novelty:** This study suggests that reactive diffusion in Cu/Zn diffusion couple occurs according to a model proposed in this paper.

Keywords: Intermetallics; CuZn System; Interdiffusion; Diffusion Couple; Reactive Diffusion

1 Introduction

Bonded dissimilar metals or metal alloys are often used particularly in the microelectronics industry. Usually, formation of one or several inter metallic compounds occurs at the interface between two substances of different chemical natures as a result of inter diffusion in the couple. This phenomenon is known as reactive diffusion and has been observed in numerous cases as Cu-Sn [1], Fe/Sn [2], Ti-Al [3], Au-Sn [4] and Ni-Sn [5]. The inter metallic compounds often have properties that are different from the base metals and excessive inter metallic compounds in a couple are detrimental [6] since they alter the mechanical [7] and electrical properties of the joint [8]. Thus, the study of reactive diffusion in binary systems is important for technological as well as for academic purposes. For technological purpose because the knowledge of the micro structure of the interfacial region and the growth kinetics of the reaction product layers may be useful for predicting the lifetime and the reliability of the joints. It is for academic purpose because the phenomenon raises questions for which there are no answers. First, when two metals A and B are put into contact, which phases of the A-B system predicted by the metals equilibrium phase diagram will form? Second, what will be the growth kinetics of the phase layer thicknesses? Third, what are the processes responsible of the phase layers growth? Zinc is a component of newly developed SnZn alloys to be used as lead-free solder [9]; the eutectic SnZn alloy (Sn-9wt.%Zn), of melting temperature (198,5°C) which is close to that of the conventional eutectic PbSn solder (183°C), is a potential candidate to be used as a Pb-free Sn-base solder in the electronics industry[10]. Copper is widely used as conductor material owing to its high electrical conductivity and good properties. When an electronic component is soldered to the Cu conductor of a circuit board, using SnZn alloy, inter metallicc compounds can form at the interface between the solder constituents and Cu metal. The inter metallic compound formation begins during the soldering process time, between the molten solder and Cu metal, and continues, at solid state, during the joint lifetime. It is of interest to determine what reaction products occur between copper and zinc in a joint containing these elements. According to the literature, only a few research groups have studied the solid-state reactive diffusion in the Cu/Zn system. A study of inter diffusion in Cu/Zn diffusion couples produced by the plating technique and then annealed at different temperatures ranging from 250°C to 380°C showed the formation of β, γ and ε phases of the Cu-Zn system at the Cu/Zn interface; it also showed that the growth kinetics of both γ and ε phase layers were parabolic at all the studied temperatures[11]. Cu/Zn couples prepared by diffusion bonding technique and then annealed at 300, 300, 350 and 380°C[12] and diffusion couples investigated at 350°C[13] indicated the formation at the Cu/Zn interfaces of only ε and γ phases. Cu/Zn couples prepared by the bonding technique and then annealed at temperatures in the range 250-350°C showed the formation of only the ε and γ phases and that the growth kinetics of both phase layers were not parabolic[14]. On the other hand, diffusion couples investigated at temperatures ranging from 290°C to 380°C showed that, at all the studied temperatures, β, γ and ε phases form at the Cu/Zn interface and grow parabolically with annealing time[15]. The discrepancies between the published works indicate clearly that the micro structure of Cu/Zn diffusion couple interface and its evolution with annealing time is still not well-known. The aim of this study was to obtain a more complete understanding of the reactive diffusion in Cu/Zn diffusion couple.

2 Methodology

2.1 Experimental procedure

In this investigation, pure Zn (99,95 at%) and pure Cu (99,99 at%), supplied by Goodfellow Cambridge Limited, were used. The diffusion couples were made with small Zn and Cu pieces about 2 and 1 mm thick respectively. In order to obtain an intimate physical contact between the couple constituents, one of the two faces of each metal piece was mechanically polished. The polishing operation guarantees not only smooth metal surfaces but also the removal of the material outer layer that could be contaminated. The metal surfaces were grounded on 600, 800, 1000, 1200, and 4000 grit emery papers, successively, and then polished by using successively 3, 1 and 0,5 μm diamond pastes. Finally, they were washed with acetone in an ultrasonic bath. Immediately after the washing, the metals were dried in warm air flux for few minutes and joined to form diffusion couples. The couple constituents were held together under an applied pressure by using a clamp. Without delay to avoid any contamination, the couple was introduced into a quartz tube which thereafter was connected to a vacuum unit and kept at room temperature until the vacuum in the tube reached 4 10^{-3} torr (5 10^{-6} atm.). Then, the tube was introduced into a tubular furnace set at the desired temperature (250°C) and kept in the furnace for one and half hour for diffusion bonding. The diffusion couple was left to cool down to room temperature, off furnace, in the evacuated tube (at 4 10^{-3} torr). After removal of the clamp, the couple was annealed at 250° C. The annealing was carried out in Argon atmosphere at atmospheric pressure. After heat treatment and cooling to room temperature, the couple was sectioned perpendicularly to the Cu/Zn interface using a Well diamond wire saw. The Cross-sections of the annealed diffusion couple were mechanically polished, as described above. The polishing process was carried out carefully, i.e., light pressure was used to avoid any damage to the micro structure of the zone of interest and the sample was thoroughly washed during the polishing in order to minimize the smearing of the materials being polished. For the same reason, the sample was polished parallel to the line of the Cu/Zn interface. In order to investigate the micro structure of the interfacial layers and determine their thicknesses, the metallographically prepared cross-sections were examined by means of a scanning electron microscope (SEM) using FEI Quanta 250 SEM equipped with secondary (SE) and back scattered electrons (BSE) detectors. The elemental chemical compositions of the formed phases were determined by energy dispersive X-ray (EDX) analysis using an EDAX-Ametek spectrometer attached to the SEM.

3 Results and Discussion

3.1 Microstructure

Scanning electron microscope (SEM) images of cross sections of Cu/Zn diffusion couples annealed at 250°C for different diffusion times are shown in Figure 1. Composition profiles of Cu and Zn elements across Cu/Zn interface obtained by EDX line scan is also shown in this Figure. The starting materials (Cu, Zn) and the reaction products can be easily distinguished on the micrographs. One can see the presence of two layers between Cu and Zn materials. It can also be noticed that the layer boundaries are practically straight lines.

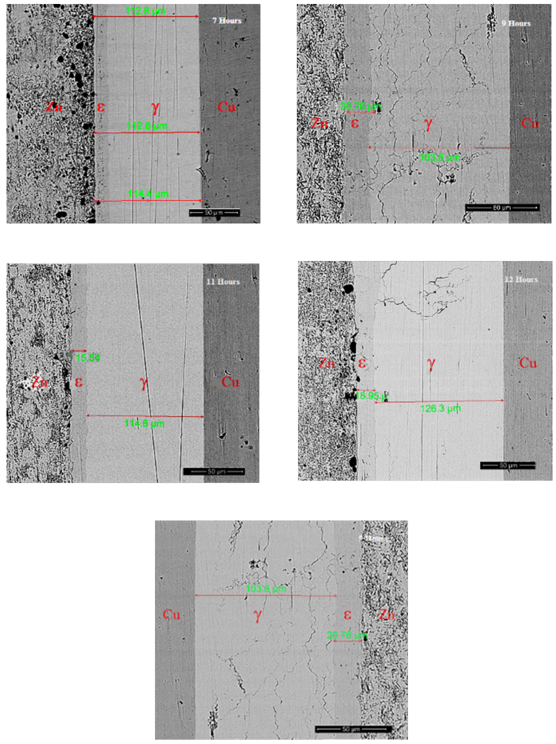

Fig 1. BSE picture of the interdiffusion zone of Cu/Zn diffusion couple for different annealing times at 250°C.

To identify the formed layers, EDX analysis was carried out in the SEM on the layers surface, at different points. According to a recent equilibrium phase diagram of the Cu-Zn system [16], three inter metallic phases are stable at the studied temperature (250°C): β, ε and γ. According to that equilibrium phase diagram, the ε and γ phases are stable for several chemical compositions around the stoichiometric ones (83,33 at % Zn) for ε and (61,53 at.% Zn) for γ. A research work done on Cu/Zn diffusion couples annealed at temperatures in the range 290-380°C indicates that the inter metallic phases possess the chemical formula CuZn, $CuZn_5$ and Cu_5Zn_8 [15], respectively. In this study, the chemical compositions, determined by EDX analysis, of the interfacial layers that formed in Cu/Zn couples after being annealed at 250 °C corresponded to the ε and γ phases of the Cu-Zn equilibrium phase diagram. The ε phase layer and the γ one was respectively on the Zn and Cu sides of Cu/Zn couple. The thickness of the ε phase layer was much smaller than that of the γ phase layer. The β phase was not detected in the couple. The absence of this phase may be due to difficulties of nucleation and/or to reactions at its boundaries canceling the total growth rate of the phase. These results are consistent with those of previous studies of solid-state reactive diffusion carried out on Cu/Zn couples isothermally annealed at temperatures in the range 250-350°C [12–14].

A SEM picture of a cross-section of a Cu/Zn diffusion couple annealed at 250° C (523 K) for 7 hours is shown in Figure 2.

One can see the location of the initial plan of contact of Cu and Zn metals. The micrograph shows clearly that both the Zn/ε and ε/γ interfaces moved towards the Zn side and the γ/Cu interface moved towards the Cu side, relative to the metals initial plan of contact. The displacements of the Zn/ε, ε/γ and γ/Cu interfaces indicate that chemical reactions took place at these interfaces. It is obvious that the variation of the thickness of the ε phase layer is due to the reactions taking place simultaneously at Zn/ε and ε/γ interfaces and that of the γ phase layer is due to the reactions occurring at ε/γ and γ/Cu interfaces. The reaction at the ε/γ interface indicates that Cu atoms arrive from Cu layer to ε layer, by diffusion within the γ layer, and form γ phase. The reaction at Zn/ε interface indicates that Cu atoms arrive from Cu layer to Zn layer surface, by diffusion within ($\varepsilon + \gamma$) layer, and form ε phase. The reaction at γ/Cu interface indicates that Zn atoms arrive from Zn metal to Cu metal, by diffusion within the ($\varepsilon + \gamma$) layer, and combine with Cu atoms to form γ phase. The change in thickness of the γ phase layer is the result of growth due to the reaction at γ/Cu interface and of that due to the reaction at ε/γ interface. That of the ε phase layer is the result of growth at Zn/ε interface and consumption at ε/γ interface. The occurrence of a chemical reaction at the Zn/ε interface, in the Cu/Zn couple, means that some Cu atoms did not react at ε/γ interface and diffused to Zn layer. The migration of the ε/γ interface from the original Zn/Cu interface towards the Zn layer is faster than that of the γ/Cu interface towards

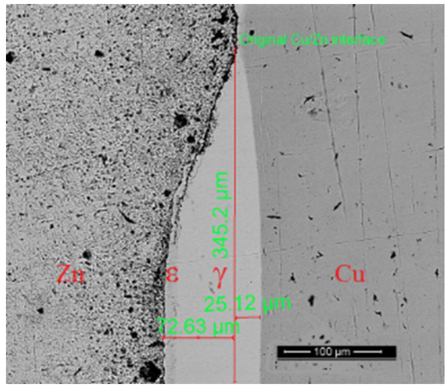

Fig 2. BSE image of a Cu/Zn cross section showing the positions of the different interfaces (Zn/ε, ε/γ, γ/Cu) relative to the initial plan of contact of Cu and Zn metals after 7 hours of annealing at 250 °C.

the Cu layer. The distance from the γ/ε interface to the Cu/Zn original interface is about 60 μm while the distance from this interface to the γ/Cu interface is around 25 μm. The ratio of the migration distances is approximately 2.4. This suggests that the γ phase layer growth is dominantly due to diffusion of Cu atoms from Cu layer towards Zn layer. Cu and Zn composition profiles obtained from a Cu/Zn diffusion couple annealed at 300°C for 28 hours have been given, indicating the positions of the different interfaces relative to the Matano plane[16]; it shows that the displacement of the ε/γ interface relative to the Matano plane is larger than that of the γ/Cu interface. This phenomenon is clearly shown in Figure 2.

On the basis of the observations made above, it is thought that reactive diffusion in a Cu/Zn couple occurs in the following way:

a) Cu atoms diffuse within γ phase from the Cu layer towards the Zn layer. Some atoms react with ε phase at the γ/ε interface to form γ phase according to the equation:

$$Cu + \frac{8}{17}CuZn_5 \rightarrow \frac{5}{17}Cu_5Zn_8 \quad (1)$$

The rest of the Cu atoms diffuse through the ε phase layer and react with Zn atoms at the Zn/ε interface to form ε phase according to the equation:

$$Cu + 5Zn \rightarrow CuZn_5 \quad (2)$$

b) Zn atoms diffuse from the Zn layer towards the Cu layer through the (ε + γ) layer and react with Cu atoms at the γ/Cu interface to form γ phase according to the equation:

$$Zn + \frac{5}{8}Cu \rightarrow \frac{1}{8}Cu_5Zn_8 \quad (3)$$

In summary, the reactive diffusion in Cu/Zn diffusion couple can be schematically illustrated in Figure 3 .

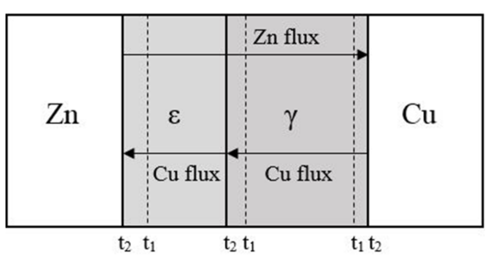

Fig 3. Schematic representation of proposed model for reaction diffusion in Cu/Zn diffusion couple; the positions of the layer boundaries at two diffusion times t_1 and $t_2 > t_1$ are shown.

3.2 Growth kinetics of inter metallic layer

To investigate the growth kinetics of the inter metallic layers in a Cu/Zn diffusion couple, several samples were prepared and annealed for different diffusion times. The thickness of the phase layers at each diffusion time was measured, in the SEM, from SEM-BSE image of the couple cross-section.

The thickness d of the γ phase layer was plotted versus the square root of diffusion time, $t^{\frac{1}{2}}$, in Figure 4 and a linear regression was fitted to the experimental data.

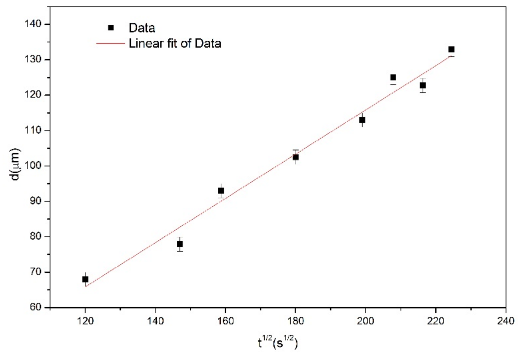

Fig 4. Plot of γ phase layer thickness,d , in Cu/Zn diffusion couple annealed at 250°C versus the square root of diffusion time $t^{\frac{1}{2}}$

As the Figure shows, the experimental points lie well on the straight line. This suggests that the γ layer thickness obeys the equation:

$$d = kt^{\frac{1}{2}} \tag{4}$$

where k is a constant. The determined value of k (= the slope of the line) is equal to $(0{,}62 \pm 0{,}035)10^{-6}\ ms^{-\frac{1}{2}}$. Theoretical works on diffusion-controlled growth of inter metallic layer in binary systems showed that the thickness-diffusion time relationship obeys the equation:

$$d^2 = 2k_p t \tag{5}$$

where k_p is called the parabolic growth constant.

The parabolic growth constant k_p of the γ layer in Cu/Zn diffusion couple, $k_p = \frac{k^2}{2}$, is then equal to:

$$k_p = (1{,}9 \pm 0{,}02)\,10^{-13} m^2 s^{-1}$$

This value is substantially smaller than the k_p value (622,8 10^{-12} m^2s^{-1}) at the same temperature (250°C) determined in a previous research work[11]. In that work, the diffusion couples were prepared by the electroplating technique. The faster growth of the Cu-Zn inter metallic layers in the electroplated Cu/Zn couples may be due to more defects (vacancies, grain boundaries) in the couples which would enhance the diffusion of Cu and Zn within the interfacial zone and hence the layers growth. In a recent research work using bulk Cu/Zn diffusion couples and annealing temperatures in the range 290- 380°C[15], the growth constant of the γ phase layer obeyed the Arrhenius equation with an apparent activation energy Q = (33,871 ± 0,976) kJ and a frequency factor k_0 = (1,375 ± 0,274)10^{-9} m^2s^{-1}. For comparison, the growth constant k of the γ layer was calculated, from these data, for the annealing temperature of the present study (250°C), which is close to 290°C, and found to be k = (5,69 ± 1,27) 10^{-13} m^2s^{-1}. However, in that work[15] the parabolic growth constant k is defined by the equation d^2 = kt while in the present study it is defined by the equation d^2 = 2 k_pt. If their data were fitted to this equation, their parabolic growth constant would be k' = k/2. Therefore, the parabolic growth constant k_p determined in this study should be compared to k/2. It is seen that the value of the parabolic growth constant determined in this study, which is given above, may be considered similar to k/2 = (2,84 ± 1,27) 10^{-13} m^2s^{-1} owing to the uncertainty on k/2.

The growth kinetics of the γ phase layer can be determined, for diffusion-controlled growth, by using Fick's first law as follows:

The flux ε/γ interface (interface 2) from Cu layer (interface 1), (see Figure 5), is given by:

$$J_1 = -D^{\gamma}_{Cu}\left(\frac{\partial c^{\gamma}_{Cu}}{\partial x}\right)_2 \tag{6}$$

where $D^{\gamma}_{Cu}$is the diffusion coefficient of Cu atoms in the γ phase layer, $c^{\gamma}_{Cu}$is the Cu concentration in the γ phase layer and $\left(\frac{\partial c^{\gamma}_{Cu}}{\partial x}\right)_2$ is the concentration gradient of Cu in this layer at interface 2.

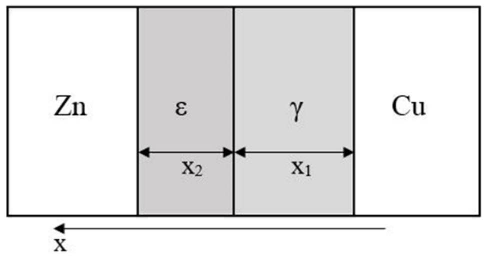

Fig 5. Schematic diagram of the interfacial region in Cu/Zn diffusion couple after a diffusion time t.

If the concentration distribution of Cu within the layer is linear

$$\left(\frac{\partial c^{\gamma}_{Cu}}{\partial x}\right)_2 = -\frac{c^{\gamma}_{Cu1} - c^{\gamma}_{Cu2}}{x_1} \tag{7}$$

where x_1 is the layer thickness and c^{γ}_{Cu1} and c^{γ}_{Cu2} are the Cu concentrations at interface 1 and interface 2 respectively. This assumption is usually made for treating the growth kinetics of compound layers[17].

Thus

$$J_1 = D^{\gamma}_{Cu}\frac{c^{\gamma}_{Cu1} - c^{\gamma}_{Cu2}}{x_1} \tag{8}$$

The flux J_2 of Cu atoms diffusing from the ε/γ interface towards the Zn layer is equal to:

$$J_2 = -D^{\varepsilon}_{Cu}\left(\frac{\partial c^{\varepsilon}_{Cu}}{\partial x}\right)_2 \tag{9}$$

where c^{ε}_{Cu} is the Cu concentration in the ε layer.

The flux J of Cu atoms reacting at the ε/γ interface with ε phase according to equation (1) is equal to:

$$J = J_1 - J_2 \tag{10}$$

Then

$$J = D^{\gamma}_{Cu}\frac{c^{\gamma}_{Cu1} - c^{\gamma}_{Cu2}}{x_1} - D^{\varepsilon}_{Cu}\frac{c^{\varepsilon}_{Cu2} - c^{\varepsilon}_{Cu3}}{x_2} \tag{11}$$

where x_2 is theεphase layer thickness at time t.

The growth rate of the γ layer thickness due to the reaction occurring at interface 2 is related to J by the equation[17]:

$$J = c^{\gamma}_{Cu2}\left(\frac{dx_1}{dt}\right) \tag{12}$$

So

$$dx_1 = \frac{1}{c^{\gamma}_{Cu2}}\left(D^{\gamma}_{Cu}\frac{c^{\gamma}_{Cu1} - c^{\gamma}_{Cu2}}{x_1} - D^{\varepsilon}_{Cu}\frac{c^{\varepsilon}_{Cu2} - c^{\varepsilon}_{Cu3}}{x_2}\right)dt \tag{13}$$

The flux$J_3$of Zn atoms reacting with Cu atoms at interface 1 is equal to

$$J_3 = -D^{\gamma}_{Zn}\left(\frac{\partial c^{\gamma}_{Zn}}{\partial x}\right)_1 \tag{14}$$

The growth rate of the γ layer thickness due to the reaction occurring at interface 1 is related to J_3 by the equation:

$$J_3 = c^{\gamma}_{Zn1}\left(\frac{dx_1}{dt}\right)_1 \tag{15}$$

and then

$$dx_1 = \frac{1}{c^{\gamma}_{Zn1}} D^{\gamma}_{Zn}\frac{c^{\gamma}_{Zn2} - c^{\gamma}_{Zn1}}{x_1}dt \tag{16}$$

The total increase of the γ layer thickness during the diffusion time dt is equal to:

$$dx_1 = \frac{1}{c^{\gamma}_{Cu2}}\left(D^{\gamma}_{Cu}\frac{c^{\gamma}_{Cu1} - c^{\gamma}_{Cu2}}{x_1} - D^{\varepsilon}_{Cu}\frac{c^{\varepsilon}_{Cu2} - c^{\varepsilon}_{Cu3}}{x_2}\right)dt + \frac{1}{c^{\gamma}_{Zn1}} D^{\gamma}_{Zn}\frac{c^{\gamma}_{Zn2} - c^{\gamma}_{Zn1}}{x_1}dt \tag{17}$$

Then

$$x_1 dx_1 = \left(D^{\gamma}_{Cu}\frac{c^{\gamma}_{Cu1} - c^{\gamma}_{Cu2}}{c^{\gamma}_{Cu2}} - D^{\gamma}_{Cu}\frac{c^{\varepsilon}_{Cu2} - c^{\varepsilon}_{Cu3}}{c^{\gamma}_{Cu2}}\frac{x_1}{x_2} + D^{\gamma}_{Zn}\frac{c^{\gamma}_{Zn2} - c^{\gamma}_{Zn1}}{c^{\gamma}_{Zn1}}\right)dt \tag{18}$$

If $\alpha = \frac{x_1}{x_2}$ is independent of time, the integration of equation (18) gives:

$$x_1 = \sqrt{2}\left(D^{\gamma}_{Cu}\frac{c^{\gamma}_{Cu1} - c^{\gamma}_{Cu2}}{c^{\gamma}_{Cu2}} - D^{\gamma}_{Cu}\frac{c^{\varepsilon}_{Cu2} - c^{\varepsilon}_{Cu3}}{c^{\gamma}_{Cu2}}\alpha + D^{\gamma}_{Zn}\frac{c^{\gamma}_{Zn2} - c^{\gamma}_{Zn1}}{c^{\gamma}_{Zn1}}\right)t^{\frac{1}{2}} \tag{19}$$

In this case, this theoretical calculation shows that the variation of the layer thickness with diffusion time is parabolic which is consistent with the obtained experimental result shown in Figure 4. α is independent of time in the case where both the 15]. In this work, the growth kinetics of the phase layer was neither parabolic nor linear.

4 Conclusion

The reactive diffusion in Cu/Zn diffusion couple at 250 °C leads to the formation of ε and γ phases of the Cu-Zn system. The phases grow in the forms of continuous layers at the interface between the metals. The growth rate of the ε phase layer is much smaller than that of the γ phase layer. The growth kinetics of the γ layer is parabolic. The study suggests that the ε layer grows as a result of diffusion of Cu atoms, from Cu layer to Zn one, and their reaction with Zn atoms; the γ layer grows, simultaneously, as a result of diffusion of Zn atoms, from Zn layer to the Cu one, and their reactions with Cu atoms, and as a result of Cu atoms diffusing from Cu layer and reacting with ε layer at ε/γ interface.

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