Study of Internal Morphology on Preparation of Cu2OThinPlate using Thermal Oxidation

by Rahadian Zainul Et.al

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Abstract. The copper oxide plate has been used as a visible light photovoltaic panel material. In this research, the process of forming copper oxide on the surface of the copper thin plate is examined. The method used is the thermal oxidation of the copper plate with a thickness of 200-250 μm. Surface evaluation and internal morphological studies of the oxide synthesis process are carried out using SEM, XRD and DTA. DTA analysis results provide information that the process of plating copper plate structure occurs at 370.44°C. XRD analysis on thin oxide plate calcined at 380°C for 1 hour, formed 92.6% Cu_2O and 6.4% CuO. Analysis of surface and morphology, the optimum oxide region formed at 25 μm thickness and internal oxygen attack reaches a maximum depth of 129 μm. The size of the copper oxide aggregate is formed in a nano multiscale particle between 300 - 800 nm.

1. Introduction

Semiconductor development has been done a lot[1-7]. Semiconductors have many advantages as materials in the cell system and advocates for the manufacture of electrochemical equipment[8].Wider applications may be used as photocatalyst materials such as metal oxides of ZnO, TiO_2 and other metal oxides. ZnO metal oxide has been used as a photocatalyst in the application of degradation and transformation of humic acid in peat water [9,10].Applications of TiO_2 nanotubes using Cu_2O for photovoltaic and photocatalytic equipment [11].

One interesting semiconductor studied today is copper oxide[12-15]. This is due to the ability of copper oxide that can work on visible light[16,17]. Rahadian et al (2015) has conducted research with the application of Cu_2O semiconductor for splitting water process in producing hydrogen gas. Copper oxide is also relatively inexpensive and not harmful to the environment [18]. The process of making and modifying the Cu_2O semiconductor is also relatively easy[19].

In previous research has been developed how the process of Cu_2O synthesis[12-15]. Chien et al. (2009) reported the synthesis and characterization of Cu_2O as p-type and AZO (Al-doped ZnO). The synthesis of Cu_2O/AZO forms a heterostructured radial nanowire [20]. Sears et al. (1984) has begun synthesizing copper oxide (Cu_2O) by thermal oxidation[21]. Through the oxidation method, the formation of Cu_2O is more easily formed and stable. It is as reported by Musa et al (1998) that at temperature 1000ºC, Cu_2O will oxidized further into CuO[22] .

Unlike previous studies, the formation of thin Cu_2O semiconductor plates has not been widely reported. Previously, the plate with a thickness of 0.32 mm was performed by Rahadian et al (2015), with the ability of the cell to reach 277,364 μWatt/m^2[8,16]. In this research, researchers investigated the process that occurred during the formation of thin plate semiconductor Cu_2O with a thickness smaller than previous studies. Surface evaluations were performed using Scanning Electron Microscope (SEM) equipment, while cross sections were performed to obtain an internal morphological study during the process of forming thin copper oxide plate.

2. Experimental Section

2.1. Tools and Materials

Tools and materials used in this research is a SEM (Hitachi S-3400N), XRD (PANalytical pw30/40), Oppo F1s Camera Aperture F-2.0 16 MP, X-Ray Fluorescence, Differential Thermal Analysis, Furnace, Analytic Tools, and Scales tools glasses. The material used in this research is the glass, Plate of Cu (PT Metalindo), acetic acid (CH_3COOH) (Merck), NaOH (Merck) and aquadest.

2.2. Methods used

2.2.1. Preparation Cuprum Plate

The copper plate used is from PT Metalindo with a thickness of 0.20 mm. Before preparing for the preparation of copper oxide thin plate, this plate was measure in thickness and washed with acetic acid and sodium hydroxide. After It was washing with acid and base, Cu plates were rinsed with aquadest, and finally dried with oven at 40-50°C for 15 minutes. Furthermore, Cu Plate was characterized by DTA, SEM and XRF. Plate cut with size 4 cm x 38.5 cm as many as 17 slabs. Before calcination, the weight of the plate will be weight to determine the weight gain after oxidation of calcination takes place.

2.2.2. Preparation of Cu_2O Thin-Plate

The preparation of Cu_2O plates was carrying out by oxidation techniques at high temperatures under oxygen conditions. The cleansed Cu plates will calcinate with the temperature of the optimization time of evaluation with DTA during optimum calcination time. The length of calcination in accordance with work procedures in previous studies reported Rahadian et al (2015), the duration of Cu calcination plate is 1 hour . Result of a plate of Cu_2O characterized using XRD, XRF and SEM.

3. Results and Discussion

DTA analysis results on Cu plates obtained at a temperature of 380°C Cu atoms began to undergo a process of structural decline. This marked a decrease at 370.44°C on the DTA chart as shown in Figure 1. In the figure, at temperature 370.44ºC a critical point was reaching where the distance between the Cu atoms on the surface of the copper plate begins to grow large and undergoes a first forging. At a temperature of 380 and above, the surface structure becomes more distant between atomic spacing so that the copper surface can attract other species of atoms in the surface area, adsorbed and eventually form the

copper oxide. Increasing the distance between atoms is what causes the change of density and atomic density of Cu atoms on the plate surface.

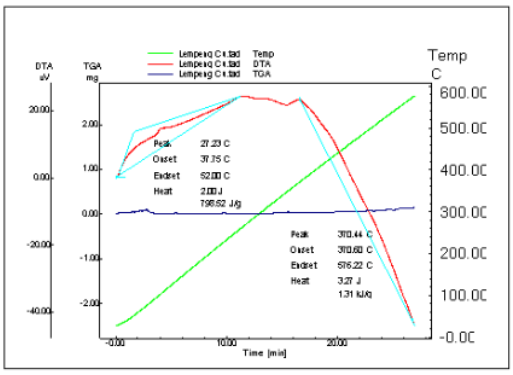

Figure 1. DTA Results for Copper Plate.

From the DTA analysis, changes in the structure of copper on the surface can predicted after 370.44-370.60 temperature was reaching. Changes in surface structures, causing migration of atoms of copper atoms into thin plate interface regions, to interact with molecular oxygen molecules. This migration process results in some oxygen atoms in the molecular state entering into the gap and the interface area expands due to the polishing of the copper plate surface. Increasing the occurrence of molecular collisions between oxygen atoms and Cu atoms, to form their oxidant compounds.

In this processing, there is a change of surface color of copper, from reddish yellow to brownish yellow and more concentrated, as shown in Figure 2. This color change indicates that there has been a change in the surface area of Cu plate. The migration of oxygen atoms from oxygen gas in the interfacial region causes the physical and chemical interactions on the surface of the copper thin plate.

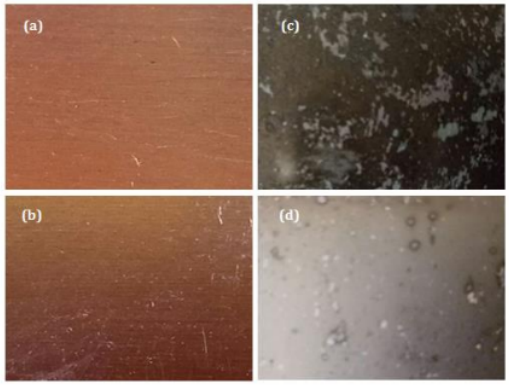

Figure 2. Copper surface photo with Aperture F Camera F 2.0-16 MP Oppo F1s at a distance of 2 cm. (A) and (b) the surface of the plate prior to calcination; (C) and (d) plate surface after calcination

The reaction is that the Oxygen molecules migrate into the surface of the copper plate, along with the spacing of the atoms between the Cu at the interface. After the occurrence of collisions between Cu and Oxygen atoms, chemical bonds occur so that the formation of copper oxide.

$Cu + O_2 \rightarrow Cu_2O$ (at temperature 400-500^oC)

$Cu_2O + O_2 \rightarrow CuO$ (at higher temperature, above 1000^oC)

The reaction of Cu_2O formation is confirmed by XRD examination as shown in Figure 3.

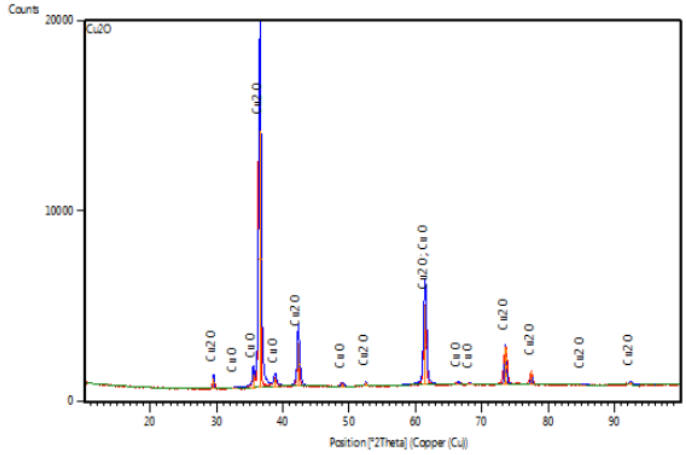

Figure 3. Results of XRD Analysis of Copper Thin Plate After Calcination

From XRD examination results, the synthesis of copper oxide thin plate can be done by calcining the plate at a temperature of 380oC. The surface change is an aggregate of oxide characterized by a peak of Cu_2O on the XRD spectra. From the result of copper oxide copper plate surface examination it is known that 92.6% oxide is Cu_2O and only 6.2% CuO. This occurs because during the process of 1 hour calcination and cooling for 4 hours, there may be oxygen atoms that are pressed and encourage further oxidation so that Cu atoms release 1 more electron. In this condition, the oxidation number of the Cu atoms on the surface of the thin plate increases from 0 to +1 and from +1 to +2. However, the percentage with continued oxidation is still relatively small.

Analysis of copper plate surfaces using Scanning Electron Microscopes (SEM) provide thermally the information that the oxidation process begins with the development or incremental distance between the surface atoms of the thin copper plate. On the surface of the copper prior to calcination, the uniformity of the atoms of Cu atoms is distributed and some parts have surface defects as shown in Figure 4. a.

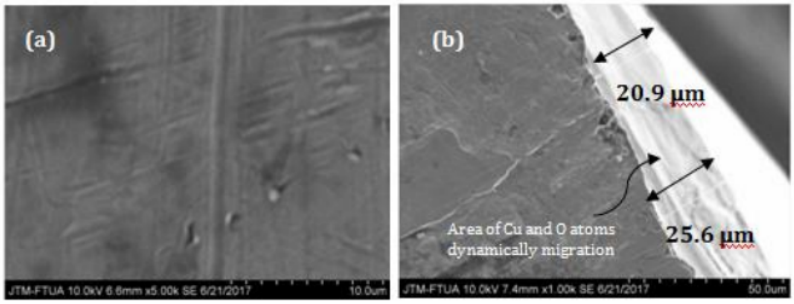

Figure 4. SEM Image of Cu Plate with 5000 x magnification (a) surface of Cu Plate with 200-250 µm thickness and (b) Internal Morphology (Cross-Section) of Cu and O atoms migration area [23]

In the internal morphology section of the plate as shown in Figure 4.b., the interface area at which the Cu atomic atoms will migrate dynamically at critical temperature points and the oxygen atom atoms can migrate into the plate surface to a depth of 20.9 µm -25.6 µm. It is in this area that the evaluation of copper oxide formation is most likely to occur as reported by rahadian et al (2015). From SEM results as shown in Figure 4.b. the oxide formation stage begins with the molecular dynamics of the interfacial region as the collisions occur between the atoms of Cu and the atomic atoms of O.

The interaction during one hour of combustion at temperature 380°C, gives weight gain on copper plate as shown in Table 1. Copper plate increased, due to the formation of copper oxide on plate surface with thickness reach 25 µm. From transverse internal SEM results, the expansion or incremental distance of Cu atoms during heating reaches the range of 50 µm, 80 µm to 129 µm. This analysis makes it possible that on Cu plates with a thickness of 200 µm - 250 µm, during calcination there has been an internal oxygen attack as shown in Figure 5.a.

In Figure 5.b. Seen several aggregates formed on the surface and on the internal oxide plate with varying sizes. Cubic and irregular shapes in multiscale with side lengths of 327 nm, 534 nm and 754 nm. This explains that the surface of the thin oxide plate formed during calcination by the thermal oxidation technique gives the size and structure of the multiscale surface, and the nano-sized particles.

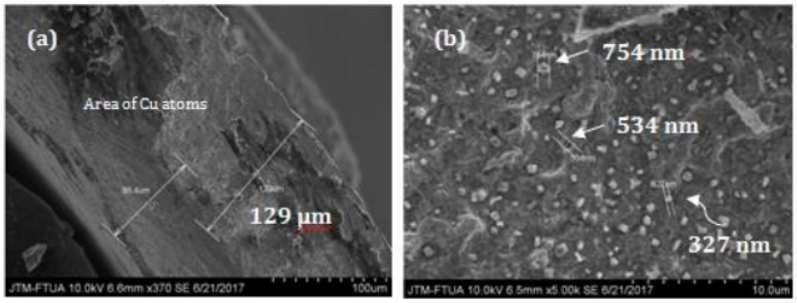

Figure 5. SEM Image of thin copper oxide plate with 5000 x magnification (a) Cross-section of Cu_2O Plate with 200µm thickness and (b) Nano Cu_2O multiscale particles

This is in accordance with previous studies that have been reported by Moses et al (1998)[22] and Sears et al (1984)[21]. In the study it was found that the formation of copper oxide began to occur at temperatures of 400°C to 1000°C[21,22]. In the first process the Cu_2O compound is formed and as the temperature rises, the Cu_2O will oxidize further to form CuO[8].

4. Conclusion

Synthesis of copper oxide on copper thin plate (thickness of 200-250 µm) was successfully performed at temperature 380oC for 1 hour. The characterization results of DTA, SEM and XRD have provided information that during thermal oxidation formed 92.6% Cu_2O and 6.4% CuO with 25 µm thickness and maximum oxygen attack at 129 µm depth. Oxides formed on the oxidation process can be applied to visible light photovoltaic panels with continued research of physical properties and optical materials to obtain materials with appropriate photovoltaic functions.

Acknowledgement

Thank you to the Government of the Republic of Indonesia, Ministry of Technology, Research and Higher Education on the Research Grand of Riset Unggulan Perguruan Tinggi in 2018-2019, and Research Grand of Terapan Universitas Negeri Padang, and Chemical Laboratory of Universitas Negeri Padang and Material Laboratory of Mechanical Engineering Department, Universitas Andalas. Thanks to the research team of UNP Chemistry undergraduate students and the Research Team from Zainul for Advanced Material Processing (ZAMP).

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