

Quantitative Analysis of Copper Sulfides by a New Electrochemical Method

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A new voltammetric method using a strongly alkaline electrolyte (6 M KOH + 1 M LiOH) as the supporting electrolyte was applied for simultaneous determination of copper oxides and sulfides. It was found that the reduction peak for Cu₂S was well separated from those of copper oxides and appeared at a slightly higher potential than that of Cu₂O. On the other hand, Cu₂O was reduced prior to the reduction of Cu₂S in 0.1 M KCl, a commonly used electrolyte in conventional methods. An electrolyte of 6 M KOH + 1 M LiOH, having a special ability to suppress the reduction of Cu₂O, enabled reversal of the reductive potentials of Cu₂O and Cu₂S. As a result, the three peaks of Cu₂O, Cu₂S and CuO were sufficiently separated. In the voltammetric measurement of a copper plate in 6 M KOH + 1 M LiOH containing S²⁻, the existence of Cu₂S was confirmed by the emergence of its redox peak pair. In addition, Cu₉S₅ was detected by its reduction peak appearing at a potential slightly higher than that of Cu₂S.

Keywords: voltammetry, 6 M KOH + 1 M LiOH, copper sulfides, copper oxides, quantitative analysis

1. Introduction

In April 2011, the authors were awarded the best paper for "Quantitative Analysis of Copper Sulfides by Voltammetry Using a Strongly Alkaline Solution"⁽¹⁾ by the Japan Society of Corrosion Engineering (JSCE). The paper was published in *Zairyo-to-Kankyo*, a journal covering techniques for corrosion and corrosion protection of metallic materials. In the paper, a new method was described that uses voltammetry to quantitatively determine copper corrosion products. The reduction mechanism was also clarified. Since both copper oxides and sulfides can be determined by a single measurement, this method can be applied for copper corrosion analysis.

2. Background of Investigation of a New Analytical Method

The color of copper surfaces changes depending on the thickness of copper oxide or copper sulfide⁽²⁾. Tarnishing frequently reduces the commercial value of industrial products. Problems, such as defective wire bonding⁽³⁾, stress corrosion cracking⁽⁴⁾ and contact resistance of connectors⁽⁵⁾, have also been reported. Each chemical species formed on copper surfaces has different properties. Therefore, qualitative and quantitative analyses of copper corrosion products are important.

The qualitative performance of surface analytical techniques, such as X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD), has been progressing in recent years. However, quantitative analysis of copper corrosion products continues to be an important task. On the other hand, in the 1930s, chemical state analysis of copper oxide films by chronopotentiometry (CP), one of various electrochemical methods, was reported⁽⁶⁾, and this method was later applied for determination of both copper oxides and chalcocite, Cu₂S^{(2), (7)}. The supporting electrolytes used

in CP measurements were neutral or weak alkaline electrolytes such as 0.1 M KCl (M = mol dm⁻³). Researchers in the field of copper corrosion have been interested in copper sulfide films in addition to copper oxide films. Recently, corrosion tests have been carried out by flowing mixed gas containing H₂S^{(8), (9)}.

In the U.S., the CP method using 0.1 M KCl for copper oxides was standardized by the American Society for Testing and Materials (ASTM). However, there is no method for copper sulfides. The contents of the latest edition⁽¹⁰⁾ are basically the same as those of the first report⁽⁶⁾. It was stated that the reduction of Cu₂O occurs first, followed by the reduction of CuO. However, no convincing evidence has been presented. Although this order is extremely important for chemical state analysis of copper oxide films, detailed re-examinations have not been carried out for more than 70 years. In recent years, the opposite viewpoint concerning the order has been reported^{(11), (12)}.

In our laboratory, copper plates having both Cu₂O and CuO films were prepared and then measured by the ASTM method. As a result, it was revealed that the separation of copper oxides was difficult. Considerable interest has been shown in the influence of copper oxides on stress corrosion cracking. Since quantitative values of Cu₂O and CuO were required, investigation of a new analytical method was needed. The method we selected was voltammetry, in which peak-like curves were obtained under appropriate analytical conditions. As a result of detailed investigation, a mixed electrolyte of 6 M KOH + 1 M LiOH as the supporting electrolyte was concluded to be the best electrolyte for selective determination of copper oxides. The research paper⁽¹³⁾ was published in the *Journal of the Electrochemical Society*. A mixed electrolyte of 6 M KOH + 1 M LiOH allowed perfect separation between two cathodic peaks due to the reduction of Cu₂O and CuO. It was also found that in the course of voltammetric reduction, CuO was first reduced to Cu in one step, followed by the reduction of Cu₂O to Cu. The reduction of Cu₂O is selectively suppressed due not only to the alkalinity of the solution but also to the ef-

fect of Li⁺ ions. In 0.1 M KCl, the reduction order was the same as the above order of CuO → Cu₂O⁽¹⁵⁾. In Japan, the Copper Alloy Data Book (1st edition) by the Japan Copper and Brass Association (JCBA) shows the reduction order of Cu₂O → CuO in CP measurements. However, the reduction order was corrected to CuO → Cu₂O in the 2nd edition⁽¹⁶⁾ using the same technical paper referenced in the 1st edition.

After the new analytical method was investigated, numerical measurements were carried out for various copper corrosion products. Although substantial results were obtained by this method, peaks at a shifted potential from that of Cu₂O were occasionally detected. Based on research of the history of such samples, detected peaks were assumed to show a reduction of copper sulfides. Investigation was also conducted focusing on the reduction of copper sulfides (mainly Cu₂S). As a result, 6 M KOH + 1 M LiOH, which was optimized for selective determination of copper oxides, could fortunately be applied for both copper oxides and Cu₂S⁽¹⁾.

At the conference presentation, there were many questions concerning the extraordinary electrolyte of 6 M KOH + 1 M LiOH. Recently, many researchers in the field of copper corrosion have been quoting our papers in Japan and overseas.

3. Summary of Analytical Method for Copper Sulfides

Details of our newly developed electrochemical method are described in a textbook⁽¹⁷⁾. Several important points concerning analytical operations are also shown in a paper⁽¹⁸⁾.

Voltammetry, a well-known method of electrochemical analysis, was applied for selective determination of copper corrosion products. There are two main analytical modes:

linear sweep voltammetry (LSV) and cyclic voltammetry (CV). In LSV measurements, the potential of copper samples was swept from the rest potential to the potential for hydrogen evolution in the cathodic direction. After that operation, in CV, the potential was swept back to the rest potential. The reduction peaks could be separated well when the sweep rate of the potential was selected appropriately for the amounts of corrosion products. The sweep rate of figures in this paper is 10 mV s⁻¹.

Electrochemical impedance spectroscopy (EIS) measurements were made over the frequency range from 10 kHz to 10 mHz with application of 1 mA DC current superimposed with 0.1 mA AC current. In the course of the measurement, copper oxide films or copper sulfide films had been reduced.

3-1 Reduction behavior of copper oxides and copper sulfides

We obtained current-potential curves for powder standard samples (i.e., Cu₂O, CuO and Cu₂S) in two different types of electrolytes. Although powder standard samples are commercially available, it is difficult to measure them directly by electrochemical methods. Therefore, prior to measurement, carbon paste was used for mixing with the powder sample⁽¹⁹⁾.

As shown in Fig. 1 A, reduction peaks for Cu₂O and CuO were clearly separated in 6 M KOH + 1 M LiOH. However, in 0.1 M KCl (Fig. 1 B), peak separation between Cu₂O and CuO was not clear. On the other hand, the reduction peak for Cu₂S was not greatly affected by the type of electrolyte. In the voltammogram for a CuS powder sample, two peaks of comparable peak areas were detected (data not shown)⁽¹⁾.

The peak for Cu₂S emerged at a slightly higher potential level than that of Cu₂O in 6 M KOH + 1 M LiOH. This order of the potential is thermodynamically abnormal. The redox reaction formulas are expressed as follows:

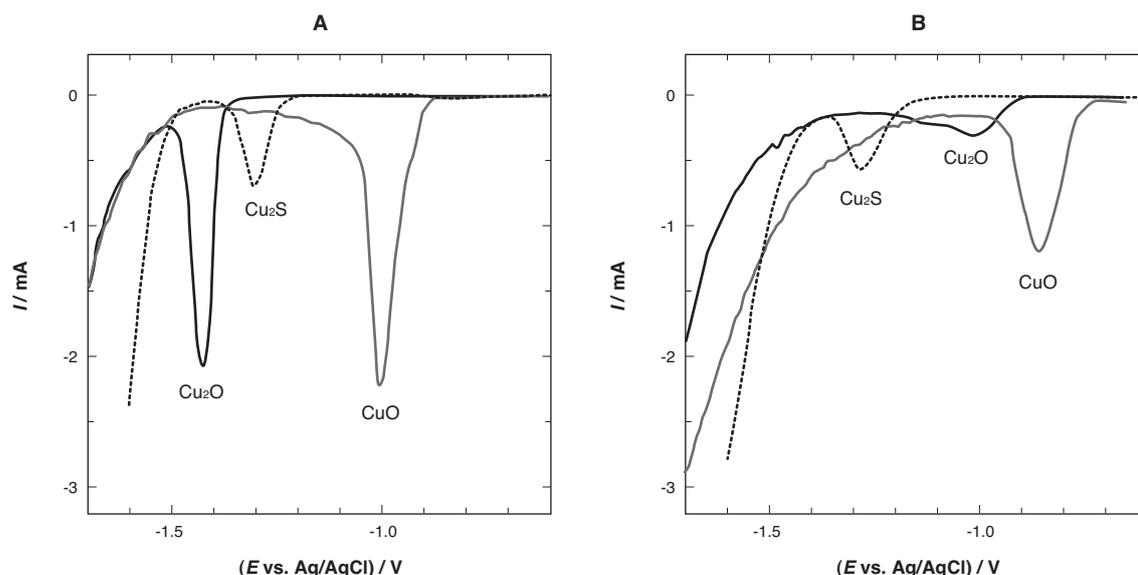
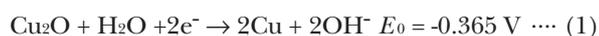


Fig. 1. Current-potential curves recorded by LSV for powder samples. Electrolyte solutions: (A) 6 M KOH + 1 M LiOH, (B) 0.1 M KCl. Scan rate: 10 mV s⁻¹



where E_0 is the normal electrode potential.

E_0 in Eq.2 has a more cathodic potential than that in Eq.1. Its value (-0.898 V) is close to the actual reduction potential (E) of Cu_2S in Fig. 1. On the other hand, E in Eq.1 depends on the pH value as follows⁽²⁰⁾:

$$E = 0.471 - 0.0591 \text{ pH} \quad \dots (3)$$

Even if a pH value of 15 is assigned to Eq.3, E shows a more positive value than -0.898 V. However, Cu_2S was reduced prior to Cu_2O . This specific behavior can not be evaluated by thermodynamical theory. On the other hand, Cu_2O was reduced prior to Cu_2S in neutral or weak alkaline electrolytes^{(2),(7)-(9)}. The reduction order in Fig. 1 B is the same as the reported one.

EIS measurements were carried out for detailed investigation of the reduction behavior of copper sulfides and copper oxides. Test samples were $\text{Cu}_2\text{S}/\text{Cu}$ ⁽¹⁾, $\text{Cu}_2\text{O}/\text{Cu}$ ⁽²¹⁾ and CuO/Cu ⁽²¹⁾ (Dumet wire: JIS H 4541, Sumitomo Electric Industries, Ltd., $\phi 0.71$ mm). The $\text{Cu}_2\text{S}/\text{Cu}$ sample was obtained by immersing the copper plates in a 5 mM Na_2S solution at 25°C for 8 h. Cu_2S is known to be formed by the reaction between copper and S^{2-} ions in an aqueous solution⁽²²⁾.

Figure 2 shows the electrochemical impedances for reductions of $\text{Cu}_2\text{O}/\text{Cu}$ and $\text{Cu}_2\text{S}/\text{Cu}$ in two types of electrolyte. These figures are called “cole-cole plots.” As seen in Fig. 2 A, the impedance behavior of Cu_2O reduction varied appreciably depending on the type of electrolyte. In 6 M KOH + 1 M LiOH, the charge transfer resistance R_{ct} (represented by the diameter of each semicircle in the

high-frequency range) was incomparably greater than that in 0.1 M KCl. Since the measurements were carried out with reduction of Cu_2O , the amplitude of R_{ct} was inversely proportional to the reduction rate of Cu_2O . That is, the reduction of Cu_2O was strongly suppressed in 6 M KOH + 1 M LiOH. In addition, an inductive loop was detected in the low-frequency range. It is generally acknowledged that the emergence of a loop suggests the presence of intermediate reaction products⁽²³⁾.

Meanwhile, R_{ct} representing Cu_2S reduction (Fig. 2 B) was practically comparable in the two types of electrolytes. This behavior is compatible with the small difference in reduction potential for Cu_2S (Fig. 1). Since the reduction of Cu_2O was selectively and strongly suppressed in 6 M KOH + 1 M LiOH, the reduction order of Cu_2O and Cu_2S was inverted depending on which electrolyte was used.

Although R_{ct} for CuO was slightly changed by the type of electrolyte, the degree of change was small compared to R_{ct} for Cu_2O . Accordingly, the ability to suppress the reduction of Cu_2O contributes greatly to the separation of Cu_2O and CuO .

3-2 Sulfidation reaction of copper

Sulfur sources are necessary for sulfidation of copper. Copper sulfides can form easily on copper surfaces when copper plates are exposed to an atmosphere containing H_2S gas or immersed in a solution containing sulfide ions.

Copper plates were first ground using SiC #2000 abrasive-coated paper and then immersed in 5 mM Na_2S for various periods. Figure 3 shows 100-magnified photos of copper samples observed with a stereomicroscope. The color of the copper surface changed in the order of red \rightarrow blue \rightarrow gray by coherency of light. Figure 4 shows current-potential curves of copper plates immersed in 5 mM Na_2S for 1, 10 and 60 min. The peak around -0.8 V to -0.9 V is the reduction peak for $\text{Cu}(\text{OH})_2$ ⁽²⁴⁾. The reduction peak increased at lower potential with increased treatment time. Besides $\text{Cu}(\text{OH})_2$, two chemical species seemed to coexist

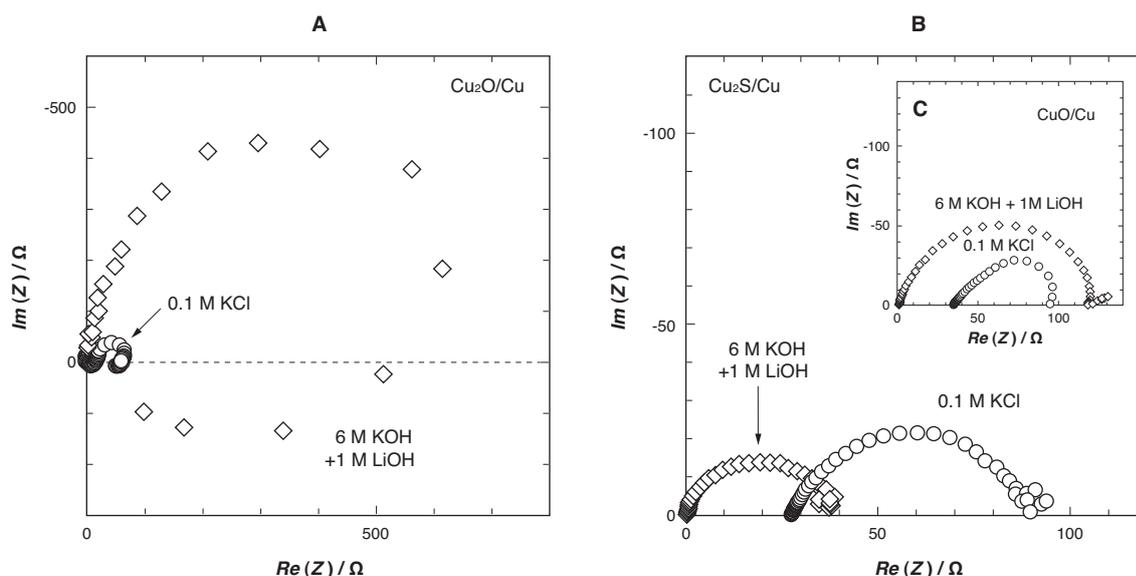


Fig. 2. Electrochemical impedances of (A) a $\text{Cu}_2\text{O}/\text{Cu}$ sample, (B) a $\text{Cu}_2\text{S}/\text{Cu}$ sample and (C) a CuO/Cu sample. Electrolyte solutions were 6 M KOH + 1 M LiOH and 0.1 M KCl. Current density: -1.0 mA cm^{-2} . Imposed current: 0.1 mA

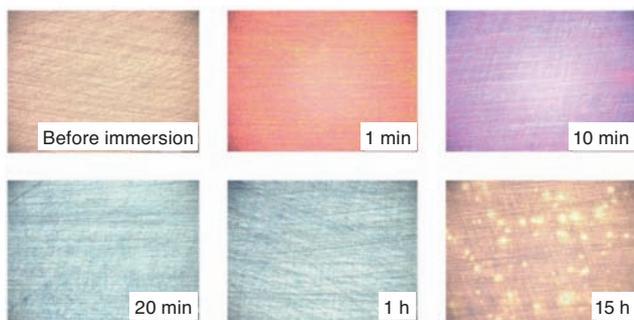


Fig. 3. Photos of copper plates immersed in 5 mM Na₂S at 25°C for various periods

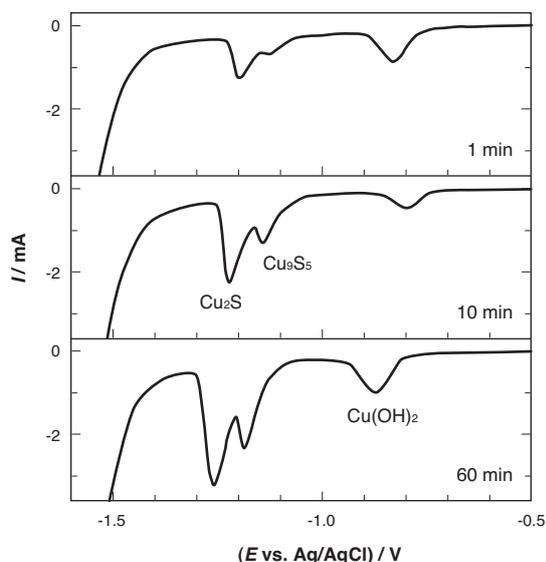


Fig. 4. Current-potential curves recorded by LSV for copper plates immersed in 5 mM Na₂S at 25°C for 1, 10 and 60 min. Electrolyte solution: 6 M KOH + 1 M LiOH. Scan rate: 10 mV s⁻¹

because this peak was split into two yielding peaks.

Figure 5 shows the XRD pattern obtained for the copper plate immersed in 5 mM Na₂S at 25°C for 8 h. A weak Cu₉S₅ phase was detected besides Cu₂S. Thus, the reduction peak at the lower potential was found to refer to a reduction of Cu₂S, whereas that at the higher potential refers to a reduction of Cu₉S₅. Using another analytical method, it is concluded that the obtained amounts of Cu₂S could be calculated using Faraday's Law and the peak area of Cu₂S based on Eq. 2⁽¹⁾. After immersing the copper plates in 5 mM Na₂S for 1 min, the average thickness of the sulfide film deposited on Cu was estimated to be around 10 nm.

Cu₂S can also be formed from Cu₂O by chemical conversion⁽²²⁾.

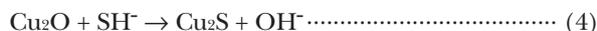


Figure 6 shows current-potential curves for CuO/Cu₂O/Cu

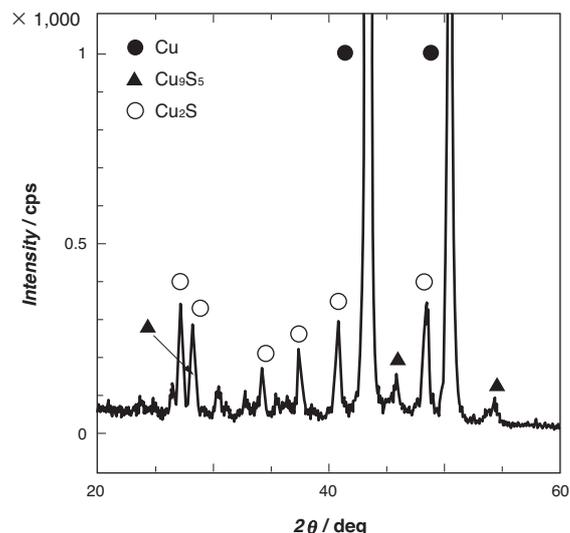


Fig. 5. XRD patterns obtained by the grazing incident method for a copper plate immersed in 5 mM Na₂S at 25°C for 8 h

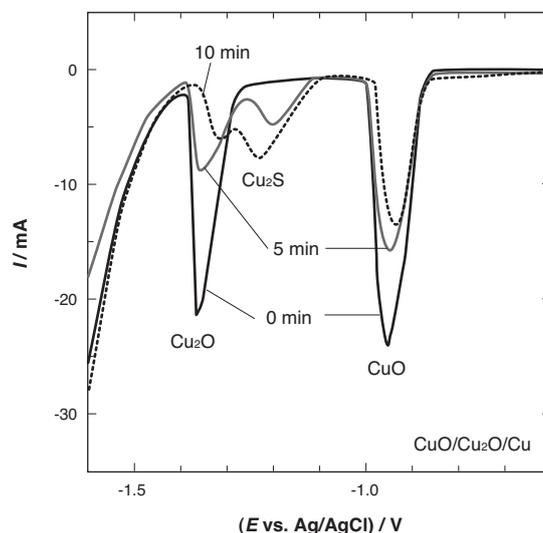


Fig. 6. Current-potential curves recorded by LSV for CuO/Cu₂O/Cu samples immersed in 5 mM Na₂S at 25°C for various periods. Electrolyte solution: 6 M KOH + 1 M LiOH. Scan rate: 10 mV s⁻¹

Cu₂O/Cu samples⁽¹⁵⁾ immersed in 5 mM Na₂S at 25°C for various periods. A CuO/Cu₂O/Cu sample has a copper film with a thickness of about 1.4 μm. In only a few minutes, both a decrease in thickness of the Cu₂O film and growth of the Cu₂S film occurred. The thickness of the CuO film also decreased in the course of immersion in 5 mM Na₂S. Thus, chemical conversion of copper oxides to Cu₂S occurred easily. CuO was assumed to have a large influence on the reaction because the rate of conversion was relatively slow in the case of using a Cu₂O/CuO sample⁽²¹⁾.

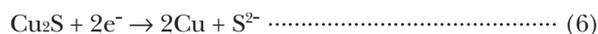
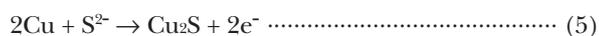
3-3 Estimation of present or absence of Cu₂S

As shown in Fig. 1 A, the reduction peaks for Cu₂O and Cu₂S emerged at nearby potential levels in 6 M KOH + 1 M LiOH. It was assumed that the distinction between

these two peaks could become difficult for copper samples with certain levels of relative ratios of Cu_2O and Cu_2S . Referring to available CV performance data reported in the case of sulfide ion injection into an alkaline solution^{(25), (26)}, similar monitoring was undertaken in 6 M KOH + 1 M LiOH to review reduction performances of Cu_2O and Cu_2S . In a high-alkaline solution, Na_2S would yield S^{2-} ions.

Figure 7 A shows current-potential curves by LSV for copper plates. A preliminary cathodic potential sweep was done from the rest potential of about -0.7 V to -1.7 V. A potential sweep was then done up to +0.7 V. In the solution containing S^{2-} ions in **7 A(b)**, an oxidation peak referring to formation of Cu_2S emerged at -1.0 V. In 6 M KOH + 1 M LiOH in **7 A(a)**, the P_1 peak was identified to represent Cu_2O formation on the top layer of the Cu surface. The P_2 peak that appeared at -0.2 V was speculated to represent the formation of $\text{Cu}(\text{OH})_2$ ⁽²⁷⁾.

Figure 7 B shows CV monitoring results for copper plates. A primary potential sweep was done from the rest potential to -1.6 V. Then, a potential sweep was done from -1.6 V up to -0.7 V and then back to -1.6 V. This operation was carried out twice. In 6 M KOH + 1 M LiOH without S^{2-} , a reduction current referring to H_2 gas evolution reaction alone was detected (**Fig. 7 B(a)**). On the other hand, in 6 M KOH + 1 M LiOH containing S^{2-} , an oxidation peak at -1.0 V (indicated by the arrow) emerged during anodic potential sweeping, and a reduction peak emerged reproducibly at -1.3 V during cathodic potential sweeping in the first and second potential sweep processes. These peaks are respectively considered to correspond to the following reactions:



That is, in the highly alkaline electrolyte containing S^{2-} , Cu_2S formed during the oxidation process and S^{2-} was given up from Cu_2S into the electrolyte during the reduction process. Under the chosen test condition, a certain level of S^{2-} was present in the electrolyte, and the intensity of the oxidation and the reduction peaks remained unchanged from the initial one in the second process of potential sweeping.

To verify the series of mechanisms shown above, CV measurement for $\text{Cu}_2\text{S}/\text{Cu}$ samples immersed in 5 mM Na_2S for 60 min was carried out. A primary potential sweep was done from the rest potential to -1.6 V. Then, from -1.6 V, a potential sweep was carried out up to the rest potential. This operation was carried out twice. As shown in **Fig. 7 B(b)**, a weak Cu_2S formation peak emerged at -1.0 V during the anodic potential sweep. The oxidation peak at -1.0 V was considered to represent the Cu substrate with S^{2-} yielded from the dissolution of Cu_2S . In the conditions used to obtain the results shown in **Fig. 8**, S^{2-} concentration near the Cu substrate was considered to be lower than in the conditions used to obtain the results shown in **Fig. 7 B(b)**. Accordingly, the intensity of the peak at -1.0 V in **Fig. 8** was weaker than in **Fig. 7 B(b)**.

By performing an anodic potential sweep after a preliminary cathodic potential sweep, the presence or absence of Cu_2S over a Cu substrate surface can be estimated. A nanometer-level thickness of Cu_2S can also be determined by separation from coexisting Cu_2O . The proposed method is useful for copper product management because the commercial value of some copper products is lost by mere tarnishing.

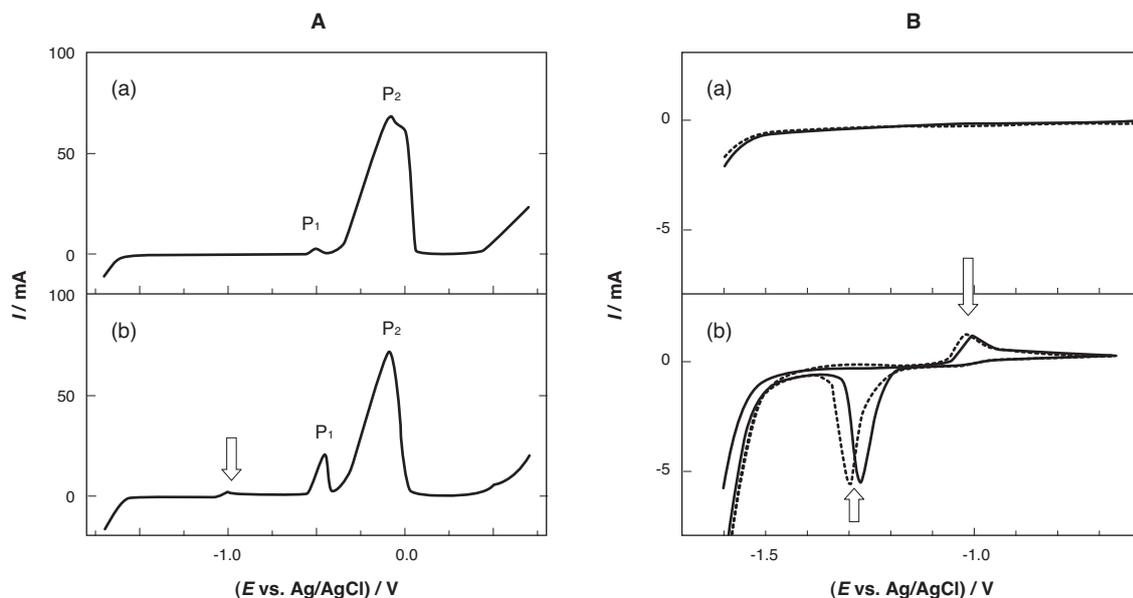


Fig. 7. (A) Current-potential curves recorded by LSV. The potential was first swept from the rest potential to -1.7 V, and then a single potential sweep was performed to +0.7 V
 (B) Current-potential curves recorded by CV. The potential was first swept from the rest potential (around -0.7 V) to -1.6 V, and then two successive cyclic potential sweeps were performed between -1.6 V and the rest potential. Electrolyte solutions:
 (a) 6 M KOH + 1 M LiOH and (b) 6 M KOH + 1 M LiOH + 5 mM Na_2S . Scan rate: 10 mV s^{-1}

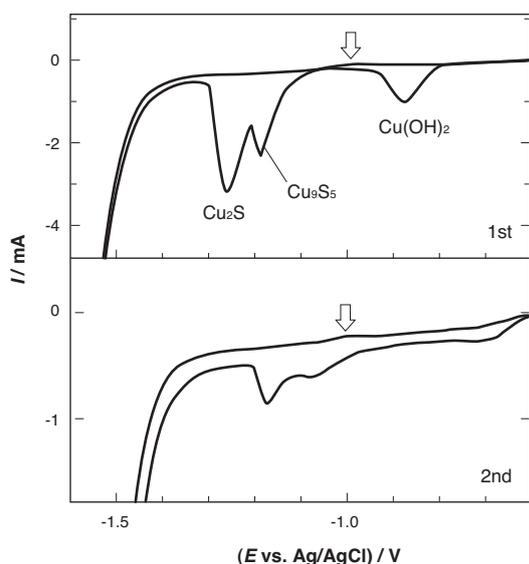


Fig. 8. Current-potential curves recorded by CV for a copper plate immersed in 5 mM Na₂S for 1 h at 25°C. Two successive cyclic potential sweeps were performed between the rest potential and -1.6 V. Electrolyte solution: 6 M KOH + 1 M LiOH. Scan rate: 10 mV s⁻¹. Arrows indicate the small anodic peak for the formation of Cu₂S on the copper surface

4. Conclusion

A new voltammetric method for estimation of copper sulfides was described. This method was investigated as an application of state analysis of copper oxides (Cu₂O and CuO). The following features concerning reduction behavior of copper sulfides were clarified.

1. The peak for Cu₂S emerged at a slightly higher potential level than the peak for Cu₂O in 6 M KOH + 1 M LiOH. This order of potential is thermodynamically abnormal.
2. Since the peak for Cu₂S emerged by sufficient separation from that of Cu₂O, the main copper corrosion products (i.e., Cu₂S, Cu₂O and CuO) were determined by a single measurement.

5. Acknowledgements

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