



## Selective flotation of chalcopyrite and molybdenite with plasma pre-treatment



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### ABSTRACT

For pretreatment of selective flotation, plasma treatment of chalcopyrite and molybdenite was applied then the minerals were washed by solution at pH 9 with oxygen bubbling. Surface characteristics of these minerals were investigated with AFM, XPS, zeta potential and contact angle measurements. Contact angle of chalcopyrite and molybdenite decreased a lot by plasma treatment. When they were washed with pH 9 solution with oxygen bubbling, contact angle of molybdenite increased whereas chalcopyrite one kept low. Adhesion force measurements indicated similar behavior. Result of flotation experiments indicated low recovery of both chalcopyrite and molybdenite after plasma treatment and only molybdenite recovery became higher after washing. Selective flotation of chalcopyrite and molybdenite could be achieved with this process. However, flotation of mixture of chalcopyrite and molybdenite after these treatments indicated both chalcopyrite and molybdenite were depressed. Addition of emulsified kerosene changed the flotation results where molybdenite was floated and chalcopyrite was depressed. Possible mechanism of selective flotation was proposed from the results of XPS, AFM, etc.

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## 1. Introduction

Molybdenite ( $\text{MoS}_2$ ) is the most important molybdenum source and large amount of molybdenite is associated with copper sulfide ore as trace mineral (Wada and Majima, 1962; Bulatovic, 2010; Cao et al., 2009; Song et al., 2012). Since molybdenite is a valuable mineral, higher recovery and efficient separation with copper sulfide is more important (Triffett et al., 2008). Conventional separation of copper sulfide and molybdenite has been achieved with sulfide bulk flotation followed by selective molybdenite flotation with NaHS addition since NaHS works as a depressant for copper sulfide (Bulatovic, 2010; Ansari and Pawlik, 2007) although imperfect molybdenite recovery (Liu et al., 2012; Zanin et al., 2009) is the issue of this process. As another selective flotation process, various oxidation methods have been proposed such as oxidation with hypochlorite (Arbiter and Young, 1951) and manganese dioxide (Curtis, 1941), oxidation with ozone (Odaka et al., 1993) is proposed recently. However, oxidation phenomenon and mechanisms are still not clear since detailed surface analyses have not done. To analyze oxidation states and evaluate mechanism can contribute

for effective separation. In this study, as a strong oxidation method for selective flotation, plasma pretreatment process was conducted. May et al. (2012) investigated the effect of plasma treatment on sulfide minerals for application to flotation. However, flotation experiments were not conducted and molybdenite was not used. Also they used plasma treatment to remove sulfur as  $\text{SO}_2$  with high energy (6000 W). We have investigated the effect of plasma with lower energy (10 W) to achieve surface oxidation. Selective flotation of molybdenite from chalcopyrite, which is the most important copper sulfide mineral, was investigated and various surface analyses were done to confirm phenomena and mechanisms of oxidation process for selective flotation.

## 2. Materials and methods

### 2.1. Materials

Pure crystals of chalcopyrite (Miyatamata mine, Japan) and molybdenite (Hirase mine, Japan) were used as experimental samples in this study. For atomic force microscopy (AFM) analysis and contact angle measurements, massive samples were prepared as following procedure. For chalcopyrite, the sample was cut as a flat shape and then it was exposed for polishing from #800 to #4000

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emery paper and Texmet (Buehler, USA) perforated non-woven pat and DP-Nap (Struers, Germany) fine polishing cloth mounted on a plate using 3  $\mu\text{m}$  and 1  $\mu\text{m}$  diamond spray. For molybdenite, the sample was cut with a scissor as a flat shape and the cleavage surface was exposed and wiped with 3  $\mu\text{m}$  and 1  $\mu\text{m}$  diamond spray. This surface was wiped by cotton with ethanol, dried with spraying nitrogen gas and repeated wiping-drying for 5 times. This surface was wiped again by cotton with pure water, dried with spraying nitrogen gas and repeated wiping-drying for 5 times.

Powder chalcopyrite sample was ground by agate mortar and screened (+75–106  $\mu\text{m}$ ). Coarse and fine powder molybdenite samples were prepared, the coarse was prepared with fine cutting followed by dry screening under 1000  $\mu\text{m}$  while the fine powder sample was supplied by Sumitomo Metal Mining, Co., Ltd., under 30  $\mu\text{m}$  and median particle diameter is 4.1  $\mu\text{m}$ . All reagents used in this study were analytical grade.

## 2.2. Plasma treatment

Plasma treatment of chalcopyrite and molybdenite sample was carried out with plasma generator (Model PA 1504, Kyoto Denshi-Keisoku Co., Ltd.) at 10 W for 1–10 min by oxygen plasma at 100 Pa using a frequency of 13.56 MHz. After plasma treatment, sample washing was achieved as following procedure: plasma treated massive and/or powder sample was suspended in a beaker with a solution of pH 9 adjusted with potassium hydroxide (hereafter; pH 9 solution) and magnetic stirred with bubbling oxygen for sample washing. After a specific period, the sample was withdrawn by filter and vacuum freeze dried for further analyses. For rest of the solution, dissolved molybdenum, copper, iron and sulfur ions were analyzed with ICP-OES.

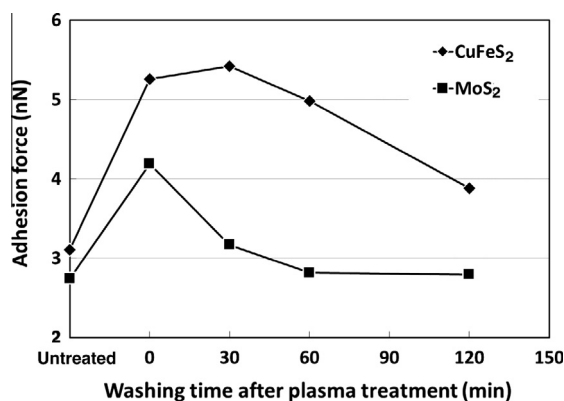
## 2.3. Atomic force microscopy (AFM)

Untreated and plasma treated samples were examined by AFM to investigate their morphology, changes in their surface characteristics and surface adhesion force. AFM images were collected in contact mode using a silicon nitride cantilever. Images were collected for each of the treated samples. Additionally, the adhesion force of the mineral was measured under ambient conditions of  $25 \pm 1^\circ\text{C}$  and relative humidity  $50 \pm 5\%$ . The force curves were then analyzed with SPIP software (Image Metrology, Denmark). Topographical images were obtained by means of NanoNavi S-image atomic force microscopy (Seiko Instruments Inc., Japan) using a

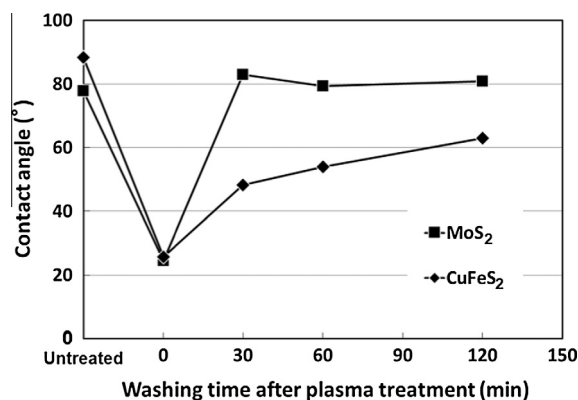
**Table 1**

Average surface roughness (nm) calculated from figure in Fig. 1.

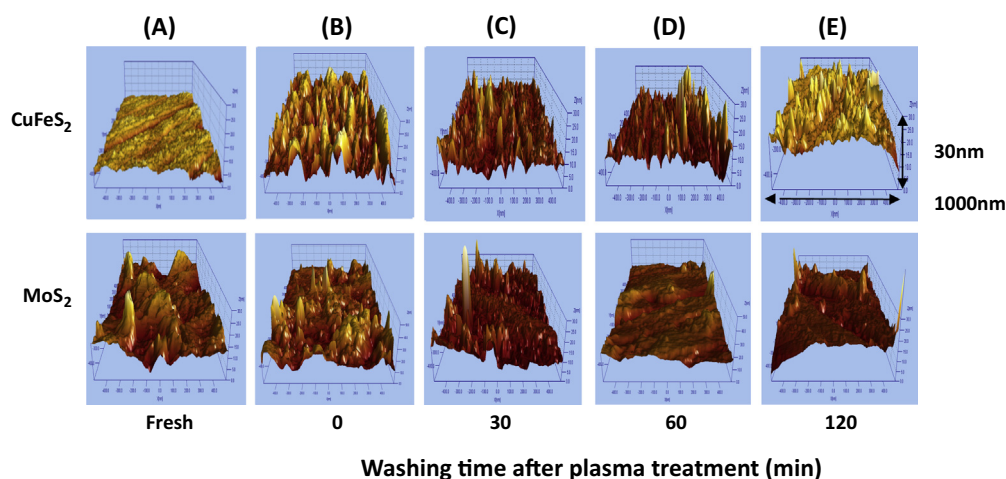
	Untreated	Washing time after plasma treatment (min)			
		0	30	60	120
Chalcopyrite	1.280	3.979	2.888	3.506	3.104
Molybdenite	3.797	5.176	4.263	3.484	3.289



**Fig. 2.** Adhesion force measurement for chalcopyrite and molybdenite with or without plasma treatment followed by washing.



**Fig. 3.** Contact angle measurement of chalcopyrite and molybdenite with or without plasma treatment followed by washing.



**Fig. 1.** AFM images of chalcopyrite and molybdenite. Untreated (A), after plasma treatment with 10 W for 10 min (B) then washed by pH9 solution for 30 min (C), 60 min (D) and 120 min (E).

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