Aplicación de secciones transversales pulidas y observación FE-SEM de relevantes generados a partir de flotación de minerales

Application of cross section polishing and FE-SEM observation to tailings generated from flotation of ores

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Resumen

Se obsevaron partículas finas en relaves generados a partir de la flotación de minerales. Mediante el uso de barrido de emisión de campo (FE-SEM), se observaron las partículas finas a nivel submicrónico con alta resolución. Para la observación de la sección transversal, las muestras de relaves fueron preparados por la sección pulida. Con la combinación de estas técnicas, se determinó la distribución de los metales pesados y metaloides en las partículas finas. Como resultado, cuando se observaron las superficies de las partículas, la detección de Pb, Sb, Fe como se indica que estos elementos coexisten o existen por separado en las partículas finas 1-3um. En la observación de la sección transversal, se encontró que las concentraciones de Zn y Fe diferente en las partículas.

Palabras clave: Relave, Metales pesados, Metaloide, FE-SEM, Sección pulida trasversal.

Abstract

Fine particles in tailings generated from the flotation of ores were observed. By using Field-emission scanning electron microscopy (FE-SEM), the fine particles were observed at submicron level with high resolution. For the cross section observation, the tailing samples were prepared by cross section polishing. With the combination of these techniques, we determined the distributions of the heavy metals and metalloids in the fine particles. As a result, when the particle surfaces were observed, the detection of Pb, Sb, Fe and/or As indicated that these elements coexist or separately exist in the $1-3$ µm fine particles. In the cross section observation, the concentrations of Zn and Fe were found to be different in the particles.

Keywords: Tailing, Heavy metals, Metalloids, FE-SEM, Cross section polishing.

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1. INTRODUCTION

Flotation is the most common method for the recovery of valuable metals from ores containing sulfides of the metals and other minerals. In the flotation, a part of heavy metals and metalloids present in the ores is transferred to the tailings, and when disposing the tailings, the soils in the disposal sites are contaminated by the heavy metals and metalloids. In fact, the soil contaminations due to the tailings were reported (Bech et al, 1997), and tailing disposal is one of the important environmental issues. To develop an effective strategy for the decontamination of the polluted sites, it is important to obtain detailed information on the tailings. Also, the concentrations of the heavy metals and metalloids in the tailings have been previously investigated. Herein, the fact that the concentrations of these elements increased with decreasing particle sizes was reported (Kim et al, 2011). Therefore, the microscopic observation of the fine particles is important to determine the heavy metals and metalloids distribution in the particles: (i) do these elements coexist or separately exist? (ii) are they present on the surface or inside of the particles?

The microscopic observation technique has been developed in the research field of advanced materials to evaluate a diversity of materials, and FE-SEM device enables the materials observation at submicron level with high accuracy and resolution. For example, the positive-electrode materials used in the lithium-ion battery consist of fine particles and their surfaces are observed at submicron level by FE-SEM (Waller et al, 2014). In addition, the cross section observation is also important in the field of the advanced materials. Recently, the graphite particles used for the electrodes in fuel battery were coated with Ni and PTFE, and their cross sections were observed to evaluate their layer thicknesses (Kim et al, 2013). For the cross section observation, particle samples are prepared by cross section polishing in which argon ion beam is irradiated to the particles to cut them. These techniques are also useful to identify the metal distribution in the tailing fine particles.

In this work, a combination of the cross section polishing and FE-SEM observation was applied to the tailing from the flotation of the ores. Our purpose is to observe and determine the distributions of the heavy metals and metalloids in the tailing fine particles. A tailing sample collected from flotation process in a practical plant was observed, and the elements present in the sample were analyzed.

II. MATERIALS AND METHODS

2.1 Materials

We have employed 500 g of the tailing generated after producing Pb, Zn and Cu concentrates in the flotation process from the treatment plant of ores provided by Milpo Group, Peru. In this processing plant, the ores from Atacocha mine located in the central highlands of Peru are treated. As known, this mine is classified into underground poly-metallic type, and the ores from the mine contains Pb, Zn, and Cu with Ag and Au content.

General information of the tailing sample is described below. Table 1 shows the chemical composition of the sample determined by X-ray fluorescence (XRF) analysis on a Shimadzu LAB CENTER XRF-1800 system (Kyoto, Japan). The main components of the sample were Ca, Si, Fe, S, Mg, and Al. Also, in the sample, SiO_2 (Quartz), $CaCO_3$ (Calcite), and FeS_2 (Pyrite) were detected by X-ray diffraction analysis on a Bruker AXS D8 ADVANCE system (Kanagawa, Japan). As can be seen in Table 1, Pb, Cu, and Zn remained in the tailing, and their concentrations were 0.046-0.75wt%. In addition, the tailing sample contained 0.067wt% of toxic As.

2.2. Analytical methods

In this study, both the surface and cross section of the particles in the tailing sample were observed. The sample preparation for cross section is explained below. Near 20 mg of the tailing sample was mixed with Epofix resin and hardener (Struers A/S, Ballerup, Denmark). The mixture was pour into a mold of 10 mm square and 1 mm thickness, which was then hardened at room temperature for 24 h. The hardened mixture with the square shape was collected, and its surface was lapped using emery paper with silicon carbide. Then, certain face of the mixture was cut by means of an argon ion beam cross section polisher on a JEOL SM-09010 system (Tokyo, Japan).

The surface and cross section of the particles were observed by FE-SEM on a Carl Zeiss ULTRA plus system (Oberkochen, Germany). This analytical system is equipped with an energy-dispersive X-ray spectrometer (EDS), and the area observed by FE-SEM was subsequently analyzed by EDS. In addition, the system has different electron detectors that provide different information on the sample (Figure 1).

III. RESULTS AND DISCUSSION

3.1. Characteristics of FE-SEM image

Before discussing the metal distributions, the characteristics of the images obtained by means of FE-SEM device are explained.

3.1.1. Images created using different detectors

Figures 2(a), (b) and (c) show the three kinds of FE-SEM images for particle surfaces in the tailing sample. As can be seen in the backscattered electron (BSE) image (Figure 2 (a)), the contrast is affected by the difference in density, and the brightness increases with increasing density. From this image, components with large and small density are identified. Figure 2 (b) shows the secondary electron image observed using Everhart Thornley secondary electron detector (E-T SE). The sample surface shape is clearly observed. Subsequently, the FE-SEM was equipped with another In-lens secondary electron detector, which detects secondary electrons with lower energy than those detected by the former. The image created using this detector is called "In-lens SE image" (Figure 2 (c)). The contrast of the image is affected by the differen-

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ce in the surface potential of the sample. In some cases, materials having different surface potentials are identified from the contrast. Thus, different types of information on the tailing are obtained by the FE-SEM system.

3.1.2. Images created under different magnifications

Figure 3 shows the E-T SE images of the surface of the tailing sample with different magnifications. As can be appreciated in Figure 3 (a), the particle sizes varied widely. The particle pointed in Figure 3 (a) was zoomed in, and the surface shape was clearly observed (Figure 3 (b) and (c)). Successively, the observation under even higher magnification was performed using the In-lens SE detector, and the obtained images is shown in Figure 4. A contrast was clearly observed on the surface as indicated by an arrow sign. As shown in the results, the fine particles in the tailing are observed at submicron level with high resolution.

3.2. Observation of heavy metals and metalloid in tailings

3.2.1. Surface of fine particles

Figure 5 (a) shows the BSE image of the particle surfaces in the tailing sample. A 1-2 μm particle with high brightness was observed as pointed by arrow, and the point analysis was performed on the particle. The spectrum of the analysis is shown in Figure 5 (b). The detection of peaks corresponding to Pb and Sb indicates that these elements coexist in the fine particle. It must be said that the concentration of Sb in the entire tailing sample was quite low and Sb was not detected by XRF as shown in Table 1. For comparison, other particles with lower brightness were analyzed by EDS, confirming that the main components of the particles were Si, Al, Mg, Ca, Fe, and O.

Figure 6 summarizes the BSE image, In-lens SE image, and spectrum of the analyzed point. In the particle with high brightness (Figure 6 (a)), Pb was present separately, not coexisting with Sb (Figure 6 (c)). Other results are shown in Figure 7. There was a 2-3 μm fine particle with high brightness (Figure 7 (a)), and As and Fe were detected in the particle (Figure 7 (c)).

3.2.2. Cross section of fine particles

Figure 8 (a) shows the BSE image of the cross sections of the fine particles. By EDS, it was confirmed that the main components of the particles were Ca and O. Also, Figures 8 (b)-(e) show the mapping images of Zn, S, Fe, and Ca obtained by EDS. The concentrations of Zn and Fe were found to be different from point to point in the particle (Figure 8 (a) and (d)).

IV. CONCLUSIONS

1. A combination of the cross section polishing and the FE-SEM observation was successfully applied to the tailing generated from the flotation of the ores. As a result, the fine particles in the tailing were clearly observed at submicron level with high precision and resolution, and the distributions of the heavy metals and metalloids in the particles were determined. We identified that Pb, Sb, Fe and/or As coexisted or existed separately in the 1-3 μm fine particles. The concentrations of Zn and Fe were different in the particles as determined by cross section observation. This information can be of great aid in designing treatment methods for toxic elements containing-tailings. Furthermore, this analytical technique is useful for the evaluation of tailings produced during metal extraction process.

V. BIBLIOGRAPHIC REFERENCES

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Table N.° 1. Chemical composition of the tailing sample.

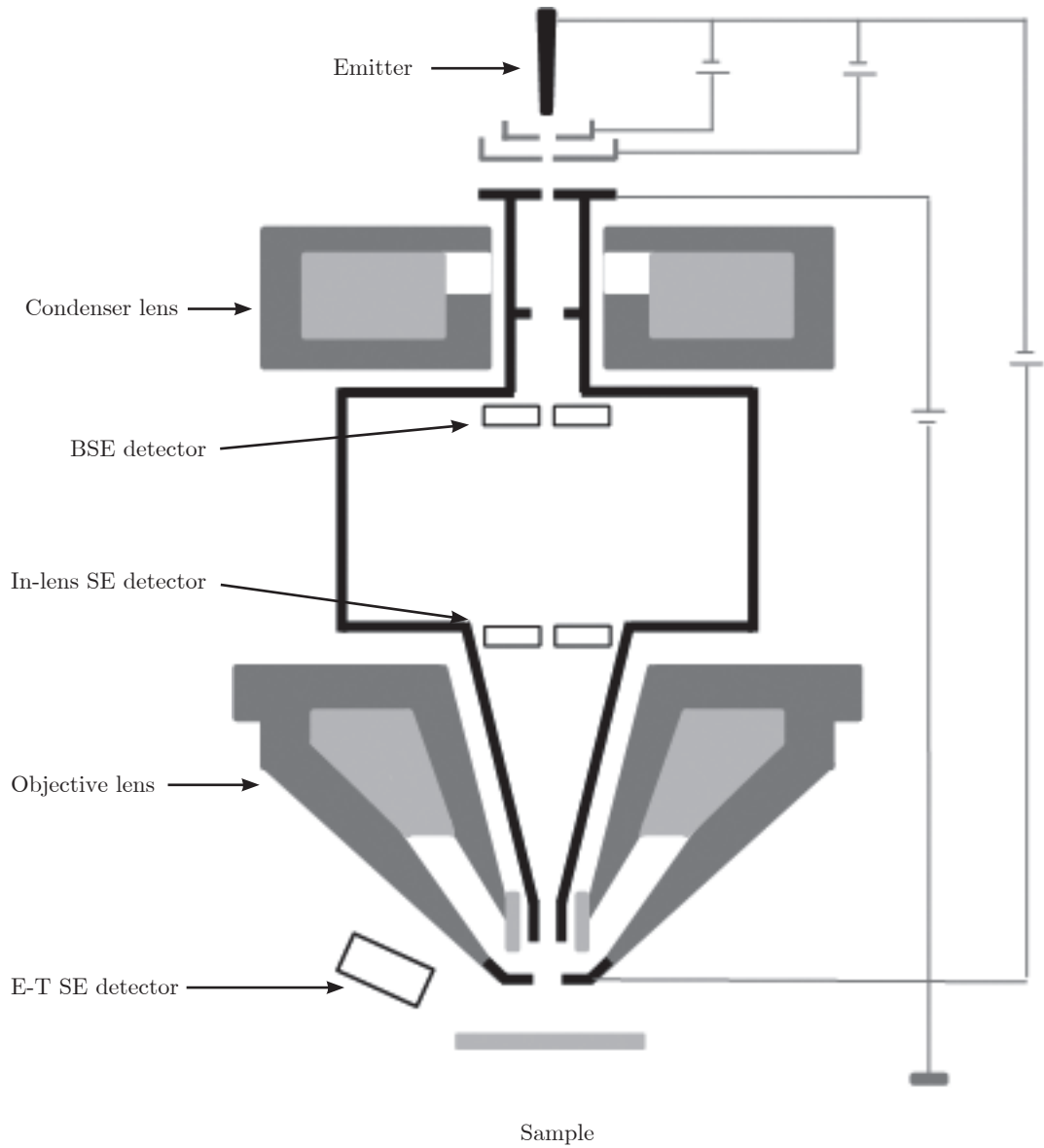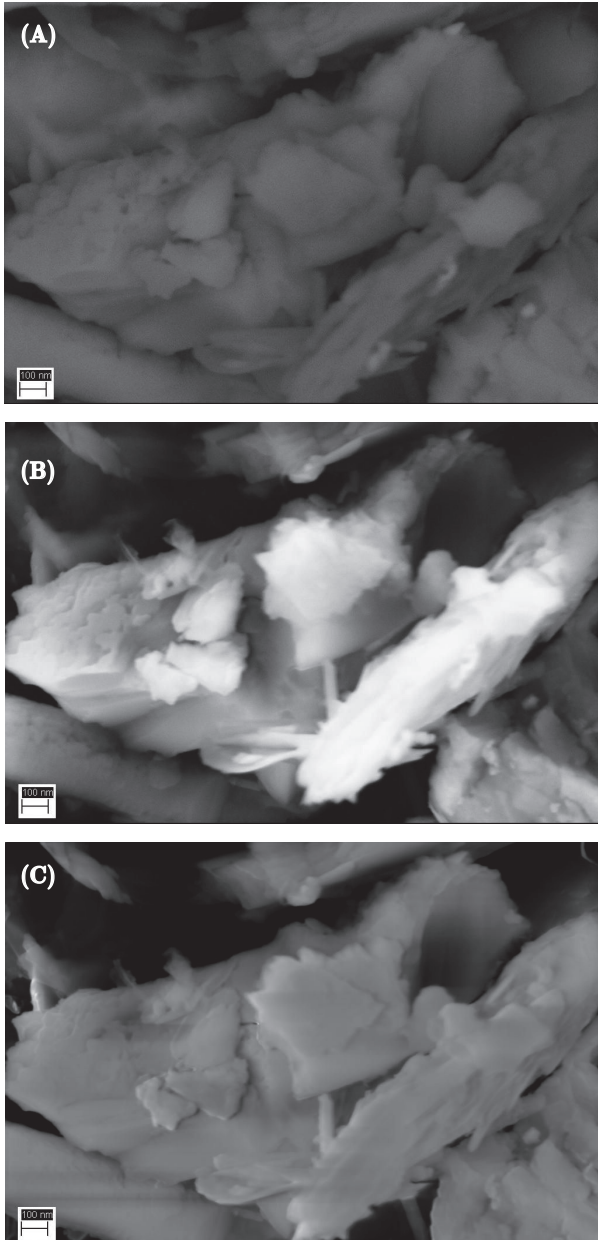|	Concentration (wt%)
SiO_2	39
CaO	27
SO_3	9.1
MgO	8.6
Fe_2O_3	8.3
Al_2O_3	4.6
K_2O	1.5
P_2O_5	0.75
PbO	0.046
ZnO	0.75
CuO	0.050
MnO	0.48
As_2O_3	0.067

Figure N.° 1. Detectors in FE-SEM device

Figure N.° 2. FE-SEM images created using the different electron detectors: (a) BSE image; (b) E-T SE image; (c) In-lens SE image.
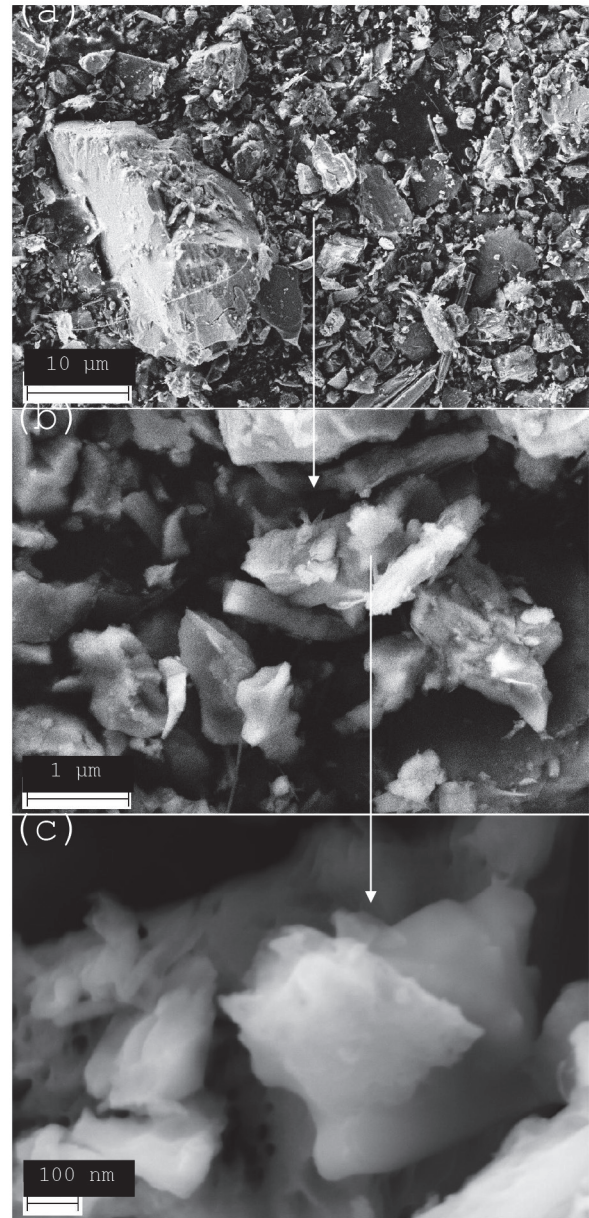

Figure N.° 3. E-T SE images created under different magnifications: (a) ×2000; (b) ×20000; (c) ×100000.
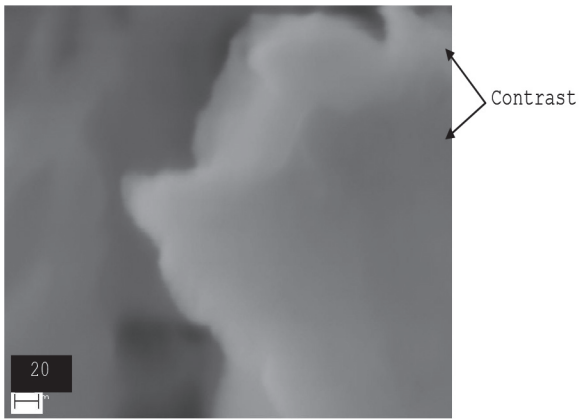
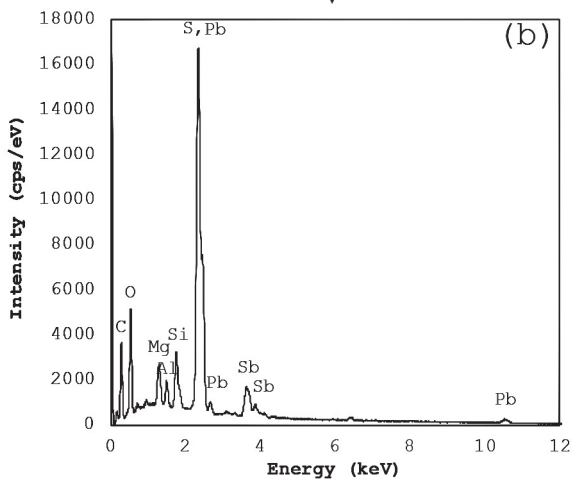**Figure N.° 4.** In-lens SE image created under high magnification.

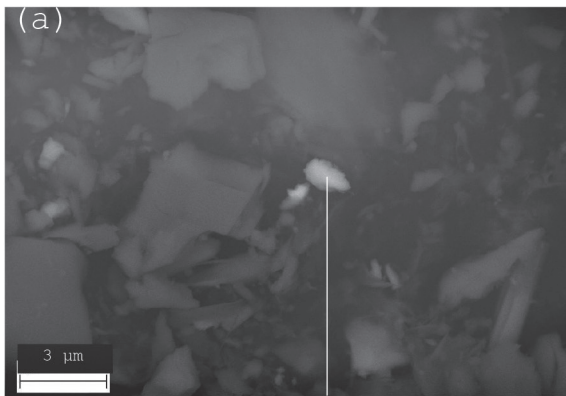**Figure N.° 5.** Pb and Sb containing-fine particle: (a) BSE image; (b) spectrum of the point analysis by EDS.
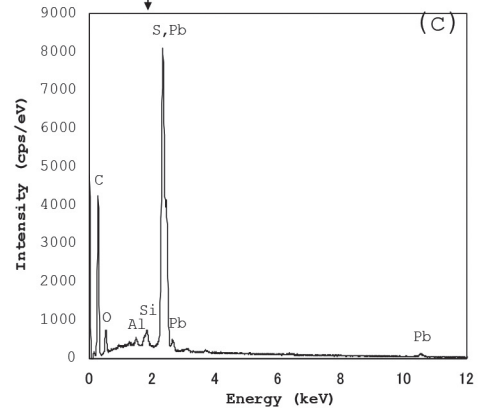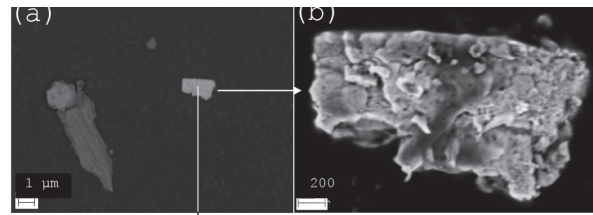

Figure N.° 6. Pb containing-fine particle: (a) BSE image; (b) In-lens SE image; (c) spectrum of the point analysis by EDS.
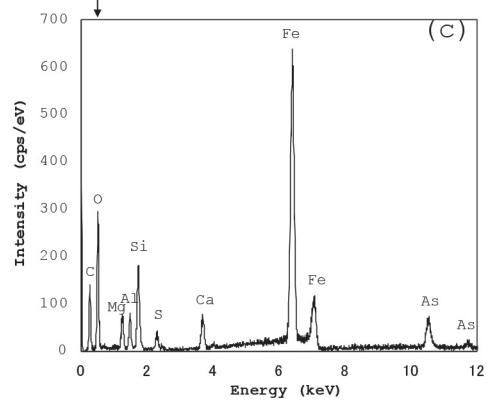

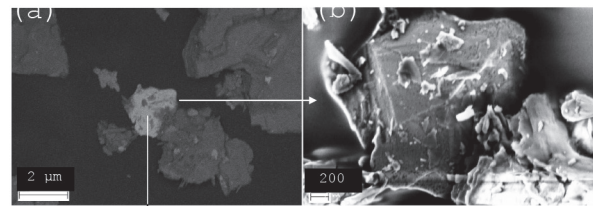**Figure N.° 7.** Fe and As containing-fine particle: (a) BSE image; (b) In-lens SE image; (c) spectrum of the point analysis by EDS.
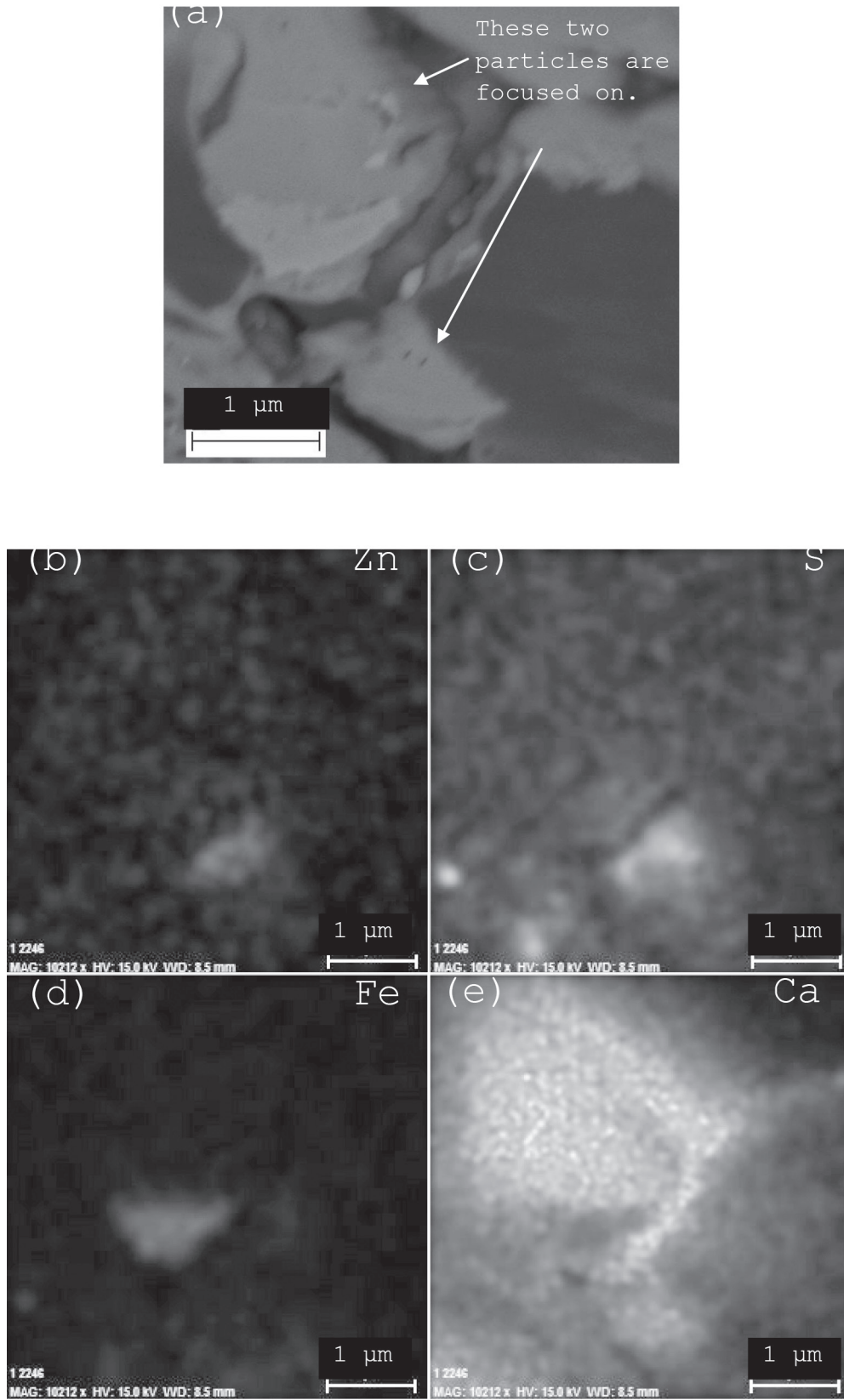
(a) These two particles are focused on.

1 µm

(b) Zn

(c) S

(d) Fe

(e) Ca

1 µm

Figure N.° 8. Cross section observation: (a) BSE image; (b)-(e) mapping images of the elements by EDS.

VI. FIGURE CAPTIONS

1. Figure N.° 1 Detectors in FE-SEM device.

2. Figure N.° 2 FE-SEM images created using the different electron detectors: (a) BSE image; (b) E-T SE image; (c) In-lens SE image.

3. Figure N.° 3 E-T SE images created under different magnifications: (a) ×2000; (b) ×20000; (c) ×100000

4. Figure N.° 4 In-lens SE image created under high magnification.

5. Figure N.° 5 Pb and Sb containing-fine particle: (a) BSE image; (b) spectrum of the point analysis by EDS.

6. Figure N.° 6 Pb containing-fine particle: (a) BSE image; (b) In-lens SE image; (c) spectrum of the point analysis by EDS.

7. Figure N.° 7 Fe and As containing-fine particle: (a) BSE image; (b) In-lens SE image; (c) spectrum of the point analysis by EDS.

8. Figure N.° 8 Cross section observation: (a) BSE image; (b)-(e) mapping images of the elements by EDS.

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