THE SIMPLE PREPARATION TECHNIQUE OF A FRAGILE SPECIMEN FOR HRTEM OBSERVATION

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ABSTRACT

The ultra thin films of fragile specimens for high-resolution transmission electron microscopy (HRTEM) observations can be successfully prepared by our simple preparation technique. At first, the specimens are impregnated with a diluted epoxy-resin (ethanol-soluble) to prevent their breakdown. Chips are made of the impregnated specimens, and these are polished. The polished chips and glasses are adhered together using an adhesive wax at ca. 70°C. The adhered chips are thinned and polished, and they form petrographic thin sections. Mo-grids are attached onto the selected areas in the thin sections for HRTEM observations using an epoxy adhesive. Only Mo-grids attached to the selected areas can be detached from the thin sections in hot water ca. 70°C. The ultra thin films are made by thinning the Mo-grids via argon ion milling.

Key words: fragile specimen, HRTEM, impregnation, ultra thin film

INTRODUCTION

High-resolution transmission electron microscopy (HRTEM) has demonstrated great ability to understand the alteration mechanism of phyllosilicates (e.g., Ahn and Peacor, 1989; Amouric and Olives, 1991; Murakami et al., 1993; Nieto et al., 1994; Shau et al., 1990). The ultra thin film of a specimen is required to be prepared for HRTEM observation. There are certain types of preparation techniques for the ultra thin film (e.g., Kogure, 1996; Vicente et al., 1997: Amouric and Olives, 1998). When microscopic observation, chemical analysis and HRTEM observation of phyllosilicates are conducted on the same point in the petrographic thin section of the specimen, the texture, chemical composition and stacking structure of phyllosilicates included in the thin section are concurrently confirmed.

It is initially required that the thin section of the specimen be prepared for such a sequence of experiments. However, the preparation of the thin section is not easy, particularly, when considerable swelling-clay minerals such as smectite, vermiculite and halloysite, which are sensitive to water and heat, are contained in the specimen. This paper describes our simple preparation technique of the fragile specimens including smectite or chlorite-smectite mixed-layer mineral for HRTEM observation.

EXPERIMENTAL MATERIALS

Our preparation technique was attempted on two different fragile specimens. One of these is weathered metabasite obtained from the Mikabu belt in Kochi Prefecture. Weathered metabasite consists of chlorite, chlorite-smectite mixed-layer mineral, smectite and actinolite (Fig. 1). This specimen is reddish brown in color and so fragile that it can be easily broken. When weathered metabasite was saturated with water, it was unable to retain its original shape.

The other specimen is hydrothermally altered pelite obtained from the Sanbagawa belt in Ehime Prefecture. Hydrothermally altered pelite consists of quartz, magnesite, dolomite, smectite and kaolinite (Fig. 1). This specimen is light greenish gray in color and is considerably harder than weathered metabasite. However, when water was dropped on the hydrothermally altered pelite, it fragmented after a period of time.

Our technique employs commercial epoxy-resin, adhesive wax and epoxy adhesive. The epoxy-resin is Eposet (Maruto Co., Ltd), which is a room temperature setting epoxy-resin. The adhesive wax is Skywax 415 (Maruto Co., Ltd), the melting point of which is 76°C.

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FIG. 1. The bulk-rock XRD patterns of experimental materials. Ethylene glycol solvation, K-saturation and heating treatment (t = 500) were also performed to identify clay minerals using oriented specimens (not shown). a) the weathered metabasite. b) the hydrothermally altered pelite. Abbreviations: chl= chlorite, chl-sm= chlorite-smectite mixed-layer mineral, sm= smectite, ka= kaolinite, ac= actinolite, qtz= quartz, mg = magnesite, do = dolomite.

It has an adhesive property of 40 kg/cm². The epoxy adhesive is standard Araldite (Nichiban Co., Ltd.).

CONVENTIONAL TECHNIQUE

At first, a dried fragile specimen is impregnated with an epoxy-resin, polyester-resin or alpha-cyanoacrylate adhesive (instant adhesive) in order to be cured. The cured specimen is cut using a diamond saw, and a chip for a petrographic thin section is obtained from it. The chip is polished with an abrasive including silicon carbide, and subsequently, with a pasty abrasive containing a diamond with a diameter of 1 μm. The polished chip and a glass are adhered together with balsam, subsequent to being heated at a temperature ranging from 110 to 120°C. A petrographic thin section is prepared for HRTEM observation by thinning and polishing the adhered chip.

Microscopic observation and electron probe microanalysis (EPMA) are performed to understand the occurrence, micro-texture and chemical composition of phyllosilicates contained in the specimen using the thin section, and suitable area to be observed by HRTEM is selected. Subsequently, the thin section is heated again at a temperature ranging from 110 to 120°C, and it is removed from the glass for the preparation of the ultra thin film. Mo-, Al- or Cu-grid is adhered using an epoxy adhesive onto the selected area in theremoved thin section. The adhered grid with the selected area is separated from the thin section and thinned to electron transparency via argon ion milling. The result is an ultra thin film for HRTEM observation made by the conventional technique.

RESULTS AND DISCUSSION

The preparation procedure of ultra thin films for HRTEM observation by our technique is compared with conventional technique in Fig. 2. Initially, the weathered metabasite and the hydrothermally altered pelite were dried for one week in a constant temperature oven at 30°C. In the conventional technique, the dried specimens were dropped into cups filled with an undiluted resin warmed slightly to reduce viscosity. Subsequently, the cups were placed in vacuum in order to be impregnated with the resin easily. In this procedure, however, the impregnation of the specimens was not attained completely. The inner portion of the specimens was not filled with the resin because the resin was still slightly viscous.

Even when the specimens were impregnated with a low-viscosity instant adhesive, the impregnation of the specimens was not sufficiently attained. The instant adhesive was immediately cured, subsequent to reacting with water contained in the specimens, and it was infiltrated into the specimens up to a depth of 1 mm. This adhesive appears to be inappropriate for specimens containing swelling-clay mineral.

In our technique, the epoxy-resin (Eposet) is diluted in ethanol to decrease its viscosity (epoxy-resin:ethanol = 1:2). The diluted epoxy-resin, which appears to be water, is dropped onto the specimens until the specimens are no longer able to absorb it. The specimens are sufficiently infiltrated with the diluted, low-viscosity epoxy-resin. Only ethanol is evaporated immediately, and the breakdown of the specimens does not occur. Consequently, micro-interstices in the specimen are filled completely with the epoxy-resin, and the complete impregnation of the specimens can be attained (Fig. 2).

Chips are obtained from the impregnated specimens using a diamond saw, and are polished. In the conventional technique, balsam was used to adhere the polished chips to the glasses, whereas in our technique, adhesive wax (Skywax) is used (Fig. 2). The polished chips and glasses are adhered with the wax, subsequent
Conventional technique

Start

The drying of specimen

The impregnation of specimen

The making of chip using a diamond saw

The polishing of chip

Balsam

At 110-120 °C (on hot plate)

Heating damage

The thin section is nonreusable

At 110-120 °C (on hot plate)
Washing in organic solvent

The detachment of thin section with heating

The adhesion of a grid on the area

The cut-out of the grid adhered on the area

Argon ion milling
Carbon coating

HRTEM observation (Fig. 5)

Our technique

Diluted epoxy-resin (Eposet)

Advantage 1. Complete impregnation

Adhesive wax (Skywax)
At ca. 70°C (on hot plate)

Advantage 2. Minimal heating damage

Microscopic observation
EPMA analysis (Fig. 3)

The selection of area for HRTEM observation

At ca. 70°C (in hot water)

The adhesion of a grid on the area

The detachment of the grid adhered on the area with heating

Fig. 2. The flowchart indicating the preparation procedure of a ultra thin film for HRTEM observation by conventional and our techniques.

to being heated at ca. 70°C. In our technique, it is unnecessary to heat the specimens over 100°C; this indicates that the change in crystal structure by heating appears to be minimized (Fig. 2). Petrographic thin sections are successfully prepared by thinning and polishing the adhered chips. Although the adhesive property of the wax is considerably weaker than that of balsam, it is sufficient for the preparation of a thin section.

Microscopic observations and EPMA analyses are conducted using the thin sections, and the suitable areas
for HRTEM observations are selected from the thin sections (Fig. 3). In our technique, Mo-grids are adhered onto the selected areas in the thin sections using the epoxy adhesive (standard Araldite). In the conventional technique, the entire area of the thin sections was detached from the glasses subsequent to being heated at a temperature range from 110 to 120°C, and these sections were not reusable. In certain cases, the detached thin section shrank and curled due to heating. Additionally, the surplus balsam is required to be extracted using organic solvents such as xylene and acetone.

In our technique, after the epoxy adhesive is cured, an incision is made in the thin section around the Mo-grids using a cutter knife. The thin sections are then placed into a plastic case filled with hot water ca. 70°C, and only Mo-grids with the selected areas can be detached from them within a few seconds. The surplus wax on the detached Mo-grids can be washed away in the hot water. In this technique, only the required area can be obtained from the thin section, as shown in Fig. 4, indicating that the thin section prepared by our technique is reusable (Fig. 2).

The detached Mo-grid is thinned to electron transparency via argon ion milling. A JEOL-2010 transmission electron microscope operating at 200 kV is used for observing lattice fringe images. A part of the observed lattice fringe images is shown in Fig. 5.

CONCLUSION

Our simple preparation technique using the diluted epoxy-resin and adhesive wax enables us to successfully prepare the ultra thin films of fragile specimens for HRTEM observations. The advantages of our preparation technique are as follows: 1) the complete impregnation of a fragile specimen can be attained, 2) heating damage to the specimens is minimized and 3) only the required area can be obtained from the petrographic thin section.

Fig. 3. Microscopic photograph (open nicol) and BSE images of the thin sections made of the impregnated specimens. a) and b) the weathered metabasite. c) and d) the hydrothermally altered pelite. See caption of Fig. 1 for explanation of abbreviations.
Simple Preparation Technique

FIG. 4. Adhered Mo-grid on thin section is indicated by black arrow. Only adhered Mo-grid can be detached in hot water (white arrow). a) the weathered metabasite. b) the hydrothermally altered pelite.

FIG. 5. HRTEM images. a) the weathered metabasite. Corrensite-like layer (ca. 2.4 nm) prevail in this image. Chlorite-like layer (ca. 1.4 nm) is also recognized. b) the hydrothermally altered pelite. Smectite-like layer (ca. 1.2 nm) is observed.

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