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**Clay mineral composition of the core sample obtained
from the bottom of the Japan Trench by TAKUYO in 1959**

Kazuo Kobayashi* and Kaoru Oinuma*

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Abstract

The core sample obtained from the bottom sediment of the northern Japan Trench was analysed clay-mineralogically. The principal clay minerals consisting of the minus-two micron fraction of the core sample are illite, montmorillonite, and chlorite.

It was confirmed that the relative amounts of these clay minerals do not show noticeable variation from portion to portion in the core sample obtained.

SPECIMEN

The core sample was collected from the bottom of the northern Japan Trench on July 4, 1959 by Takuyo, the observation ship of Maritime Safety Board. The locality of the sample and the water-depth are as follows:

Latitude	37° 29' N
Longitude	143° 53' E
Water depth	7065 meters

The core sample, about 36 cm in length, is grayish yellow to brown, and clayey. It was divided into three parts, upper (11 cm in length), middle (7 cm), and lower (18 cm) parts. Clay mineralogical analysis was carried in each of them.

The procedure for identification of clay minerals are as follows.

I. X-ray analysis

1) Randomly oriented powder

* Geological and Mineralogical Institute, Faculty of Science, Tokyo University of Education

- a) Raw sample
- b) Clay fraction less than two microns.
- 2) Well oriented aggregate of Clay fraction less than two microns,
 - a) untreated.
 - b) heated at 300°C, 450°C and 600°C.
 - c) treated with ethylene glycol or glycerol.
 - d) treated with ammonium nitrate solution (1N).
 - e) treated with magnesium acetate solution (1N).
 - f) treated with hydrochloric acid (6N).

II. Electron micrographic observation

Heat treatments: Silica glass-slide was used in the case of heat treatments. Oriented aggregates on this slide were heated at every temperature as 300°C, 450°C, and 600°C for 45 minutes, air quenched, and immediately examined by X-ray.

Treatment with ethylene glycol or glycerol (Walker, 1957, 1958) for detection of expanding lattices: Clay powders on the glass-slide were saturated by these reagents, and a wet clay film was made, in which, usually, preferred orientation of clay particles was broken. Hence, before radiating X-ray on the film, for the purpose to bring the wet film into well-oriented aggregates, it was spread over the glass-slide by rubbing the film by the surface of another glass-slide.

Treatment with ammonium nitrate solution for detection of Mg-vermiculite (Walker, 1949): The sample was boiled for 10 minutes in 1 N solution, washed with distilled water, and oriented aggregate was made.

Treatment with magnesium acetate (Rolf and Jeffreis, 1952) to know the degree of weathering of illite: Oriented aggregate was made with sample boiled in 1 N solution for one hour and washed with distilled water.

Treatment with hydrochloric acid: The sample was heated at 95°C in 6 N hydrochloric acid for 10 minutes, 30 minutes, and one hour, and washed with distilled water. Effects of this treatments were studied on some standard specimens of kaolinite and chlorite (Table 1). As shown in Table 1, it can be said that, usually, chlorite is more easily decomposed than kaolinite, and this treatment is useful for discriminating kaolinite from chlorite in mixture of these minerals.

RESULTS

X-ray analyses

X-ray diffraction patterns of randomly oriented powders of the raw sample and the clay fraction less than two microns are shown in Plate I-1.

In the case of the raw material, the X-ray reflection peaks due to quartz and feldspar were clearly confirmed, but the reflections of clay minerals were

too weak to identify them. In the case of the clay fraction, the reflections at 15.2 Å, 10.2 Å, and 4.51 Å of clay minerals were confirmed along with quartz and feldspar lines which were weaker than in the case of the raw sample.

Identification of each mineral component is difficult from the above data, because the 15 Å reflection suggests the possibility of the presence of montmorillonite or chlorite or both. For the purpose of identification of each clay mineral component, the writers investigated the effects of the following treatments upon X-ray diffraction data of well-oriented aggregates of sample powders.

(1) Upper part specimen of the core sample (Plate I-2 and Table 2)—

Treatment with ethylene glycol: The 15.8 Å reflection was replaced by a broad reflection at about 17.7 Å. It is doubtful whether the 15.8 Å reflection was entirely replaced by the 17.7 Å one because of the broad reflection at about 17 Å; In some specimens, the 17 Å broad peak occurs associating with the 15 Å one, and in some others, the 15 Å peak is not detected. Even in the latter case, however, we could not exclude the possibility that the 15 Å peak was masked in the broad 17 Å one, which usually shows an asymmetrical tailing toward a large angle-side. This fact suggests that the 15.8 Å reflection is largely due to an expanding lattice.

Heat treatment: The 15.8 Å reflection was weakened, and its spacing decreased, whereas the 10.3 Å reflection became slightly stronger after heating at 300°C and 450°C. This fact suggests the presence of a contractable lattice by heating. After heating at 600°C, the 15.8 Å spacing further decreased to 13.8 Å, and the 7 Å reflection was disappeared. These facts show the presence of chlorite. It has been reported by many workers that the chlorite in sediments usually loss diffraction effect after heating at a lower temperature comparing with well crystallized sorts of hydrothermal origin (John, Grim, and Bradley, 1954, Soveri, 1956, Dalton, Swinford, and Jewety, 1958 Oinuma, Kobayashi and Sudo 1959, Kobayashi and Oinuma, 1960, Kobayashi, Oinuma and Sudo, 1960).

Treatment with ammonium nitrate solution: The 15.8 Å reflection was replaced by the broad reflection extending from 14.3 Å to 12.6 Å. This treatment seems to give no noticeable effect on the 10 Å peak. Usually it has been reported that, by this treatment, vermiculite contracts to the 10 Å-lattice (Walker, 1949), whereas montmorillonite shows a partial contraction to about the 12 Å-lattice. Hence the result obtained from the present specimen suggests that the presence of a large amount of vermiculite was not expected. Thus the expanding lattice and the contractable lattice noticed above can be considered to be attributed to montmorillonite.

Treatment with magnesium acetate: This treatment does not give any effect on the 15.8 Å and the 10.3 Å reflections. This fact shows that the illite has not "degraded" attribute.

Treatment with hydrochloric acid: The 15.8 Å and 7.2 Å reflection were almost disappeared. This fact implies that kaolinite is absent in the specimen or even if it is present, its amount is too small to be detected by X-ray. Conclusively, the clay minerals in this specimen can be enumerated as montmorillonite, illite, and chlorite.

(2) Lower part specimen of the core sample (Plate I-3 and Table 3)—

The results are nearly same as those of the upper part, and show that the clay minerals in the specimen are designated as montmorillonite, illite, and chlorite.

Identification of kaolinite in the mixture of clay minerals used by the (060) reflections: The (060) line of kaolinite appears usually at 1.49 Å, but chlorite, illite, and montmorillonite at 1.50-1.56 Å. Thus it is expected that the presence of kaolinite can be confirmed from the (060) spacing, but, usually, it is very difficult to detect a small amount of kaolinite.

Fig. 4 shows the reflections in the range of 1.45-1.59 Å of the present specimens and standard kaolinite specimen. The absence of the 1.49 Å peak in the spectrograms of the present samples still supports the above conclusion that kaolinite is absent in the present specimen, or even if it is present, its amount would be very small.

X-ray spectrograms of the test powders were compared with one another in Plate I-5. They were obtained under the same experimental conditions; X-ray was radiated under a fixed instrumental condition over a clay film containing a fixed amount of test powders and spreading over a fixed area on the glass-slide. As shown in Plate I-5, the relative intensities of peaks do not show noticeable variation through these specimens. This fact suggest that the relative amount of clay mineral also is not in noticeable variance.

Electron micrographic observation

Electron micrographs of the sample were taken by shadowing with chromium (Pl. II, III, IV). These photographs consists of clay minerals with irregular shapes, amorphous materials, and fragments of diatom. It is difficult to confirm the kind of the clay minerals from the morphological difference.

CONCLUSION

The principal clay minerals in the clay fraction of the present specimen are enumerated as montmorillonite, chlorite and illite. This clay mineral composition of the sample is similar to that of the sample collected from the Ramapo Deep in the southern Japan Trench. (Kobayashi, Oinuma and

Sudo, 1960). There is no noticeable differences among three specimen, upper, middle, and lower, with respect to the relative proportion of those clay minerals.

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TABLE 1. EFFECTS OF TREATMENT WITH HYDROCHLORIC ACID (6 N)
ON THE 7 Å REFLECTION OF KAOLINITE AND CHLORITE

Sample	Untreated	Time of treatment				
		10 minutes	30 minutes	1 hour	2 hours	3 hours
Kampaku kaolinite	1.0	>1.0	>1.0	>1.0	>1.0	>1.0
Kaolinite	1.0	>1.0	>1.0	1.0	0.8	0.4
HS 6 kaolinite	1.0	>1.0	>1.0	>1.0	1.0	0.9
Zao halloysite	1.0	>1.0	>1.0	0.9	0.9	0.6
Hitachi chlorite	1.0	>1.0	>1.0	0.6	0.2	—
Leuchtenbergite	1.0	0.4	0.1	—	—	—
Chamosite	1.0	—	—	—	—	—

Intensity of the untreated specimen is placed as 1.0.

Kampaku kaolinite; from the Kampaku mine, Tochigi Prefecture, Japan.

Kaolinite; from the Hara district, Gifu Prefecture, Japan.

TABLE 2. X-RAY DIFFRACTION DATA OF THE UPPER PART SPECIMEN AFTER VARIOUS TREATMENTS

Untreated		300°C		450°C		600°C		Ethylene glycol		Ammonium nitrate		Magnesium acetate		Hydrochloric acid					
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	10 minutes		30 minutes		1 hour	
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I
								17.7	9										
15.8	25	14.5	7			13.6	6			14.2	13	15.2	19	14.7		5b			
										12.6				13.3					
10.3	13	10.2	19	10.3	26	10.2	20	10.2	7	10.2	11	10.2	10	10.2	14	10.2	11	10.2	11
7.2	13	7.2	15	7.2	10			7.1	6	7.1	11	7.2	15						
5.04	5	5.01	7	5.04	7	5.01	9	4.98	4	5.04	4	5.04	5	5.04	5	5.01	4		
4.77	4	4.75	3	4.75	4							5.00	4						
4.48	4	4.51	7	4.53	7	4.53	8	4.51	4	4.48	9	4.51	7	4.48	7	4.48	5		
4.27	9	4.27	10	4.29	9	4.25	8	4.29	6	4.27	13	4.27	9	4.29	3	4.27	12		
4.06	5	4.06	6	4.08	7	4.05	6			4.06	9	4.06	6	4.06	3	4.06	7		
								3.79	6					3.79	4				
3.56	7	3.56	6	3.56	7			3.55	3	3.53	7	3.56	7						
3.36	38	3.35	39	3.36	40	3.35	42	3.36	14	3.35	7	3.35	35	3.35	40	3.35	54		
3.22	10	3.21	11	3.22	9	3.21	12			3.20	14	3.20	10	3.22	9	3.20	11		

b: broad

TABLE 3. X-RAY DIFFRACTION DATA OF THE LOWER PART SPECIMEN AFTER VARIOUS TREATMENTS

Untreated		300° C		450° C		600° C		Ethylene glycol		Ammonium nitrate		Magnesium acetate		Hydrochloric acid							
d (Å)		I		d (Å)		I		d (Å)		I		d (Å)		I		10 minutes		30 minutes		1 hour	
d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I	d (Å)	I
15.8	21	14.3	9	14.0	5	14.3	11	17.0	9	14.3	18	14.7	19	14.3	2	14.5	2b				
10.2	16	10.2	18	10.2	29	10.2	32	10.2	8	10.2	20	10.2	13	10.3	9	10.2	9	10.2	9		
7.2	16	7.2	15	7.2	11			7.1	8	7.1	16	7.2	15								
				6.5	3																
5.09	7	5.04	5	5.01	8	5.04	8			4.98	6	5.04	5	5.01	5	5.01	5				
4.75	2	4.77	5	4.72	5					4.75	5										
4.51	6	4.53	5	4.48	9	4.51	9	4.48	8	4.51	8			4.48	5	4.48	7				
4.27	7	4.27	7	4.27	12	4.27	9	4.27	8	4.27	9	4.27	7	4.27	11	4.27	12				
4.06	6	4.04	5	4.06	7	4.08	5	4.04	8	4.07	6			4.06	7	4.06	9				
3.79	7					3.79	5			3.80	6			3.78	5	3.77	7				
														3.69	4	3.66	5				
3.56	7	3.56	7	3.58	8					3.55	11	3.55	7	3.51	4	3.51	4				
3.35	35	3.35	36	3.35	42	3.35	45	3.35	25	3.34	58	3.35	38	3.35	53	3.35	70				
3.22	6	3.22	8	3.22	12	3.21	11	3.20	5	3.21	14	3.20	10	3.21	10	3.20	13				

b: broad

CLAY MINERAL COMPOSITION

HS 6 kaolinite; from the Kamisunagawa district, in Hokkaido, Japan (Kobayashi and Oinuma, 1960).

Zao halloysite; from the Zao mine, Yamagata Prefecture, Japan.

Hitachi chlorite; from the Hitachi mine, Ibaragi Prefecture, Japan.

Leuchtenbergite; from the Wanibuchi mine, Shimane Prefecture, Japan.

Chamosite; from the Arakawa mine, Akita Prefecture.

— means that the peak was disappeared after this treatment.

>1.0 means that the intensity of the reflection after this treatment was larger than that of untreated sample.

Explanation of Plates

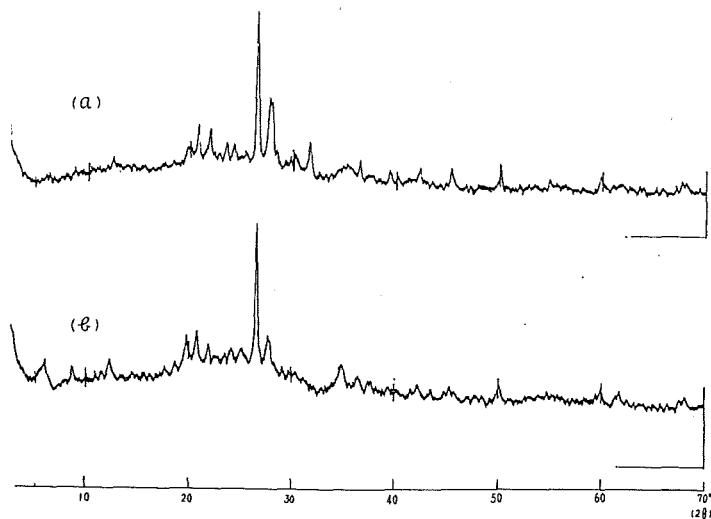
Plate I. X-ray diffractometer curves.

1. X-ray diffractions of randomly oriented powder of the middle part specimen of the core sample.
 - (a) Raw sample.
 - (b) Clay fraction less than two microns.
2. X-ray diffractometer curves of the upper part specimen after various treatments.
 1. Untreated.
 2. Heated to 300°C.
 3. Heated to 450°C.
 4. Heated to 600°C.
 5. Treated with ethylene glycol.
 6. Treated with ammonium nitrate.
 7. Treated with magnesium acetate.
 8. Treated with hydrochloric acid for 10 minutes.
 9. Treated with hydrochloric acid for 30 minutes.
 10. Treated with hydrochloric acid for one hour.
3. X-ray diffractometer curves of the lower part specimen after various treatments.
 1. Untreated.
 2. Heated to 300°C.
 3. Heated to 450°C.
 4. Heated to 600°C.
 5. Treated with ethylene glycol.
 6. Treated with ammonium nitrate.
 7. Treated with magnesium acetate.
 8. Treated with hydrochloric acid for 10 minutes.
 9. Treated with hydrochloric acid for 30 minutes.
 10. Treated with hydrochloric acid for one hour.
4. X-ray reflection patterns in the region 1.45-1.59 Å of the specimens and kaolinite sample.

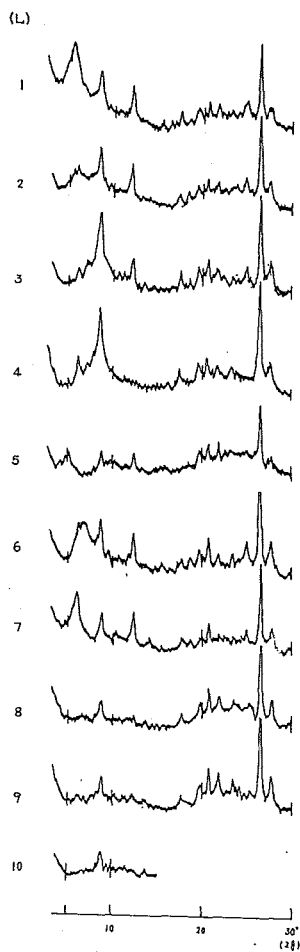
The patterns were obtained from the randomly oriented powders of specimens. In the patterns the (060) reflections of clay minerals are seen.

Plate I. Clay mineral (Kobayashi and Oinuma)

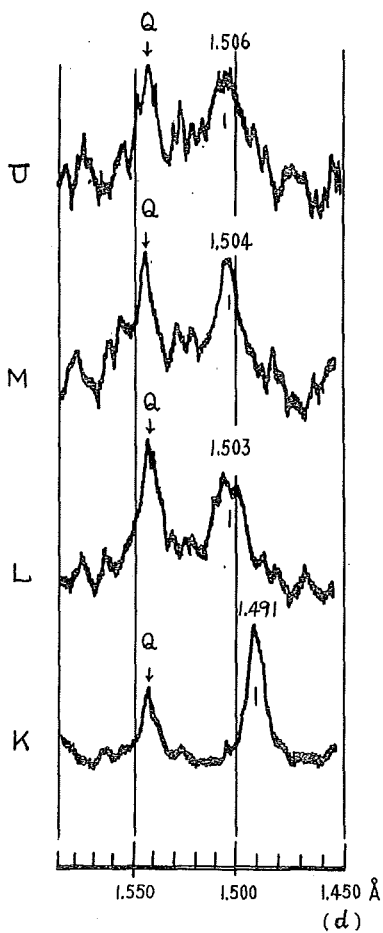
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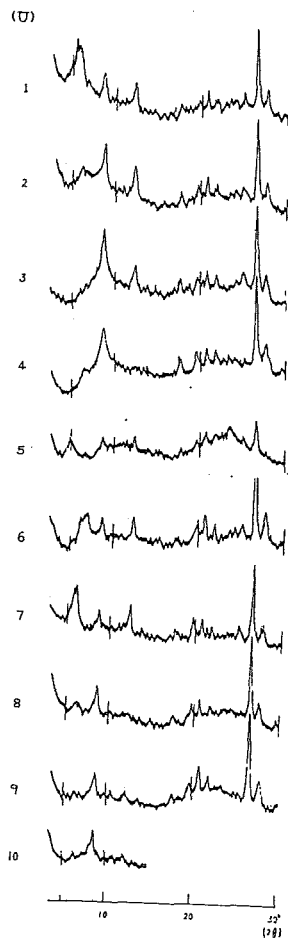
3



4



2



5

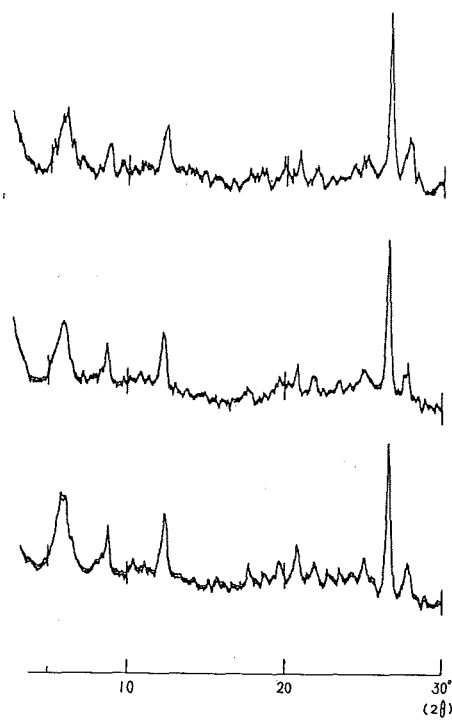


Plate II. Clay mineral (Kobayashi and Onuma)

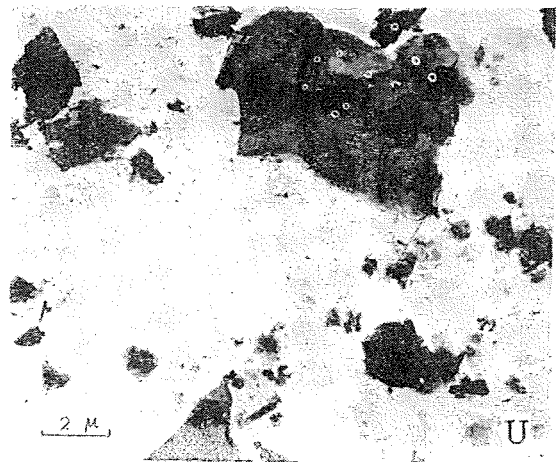
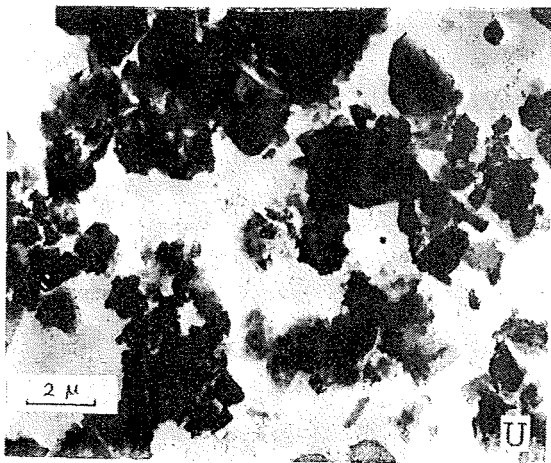
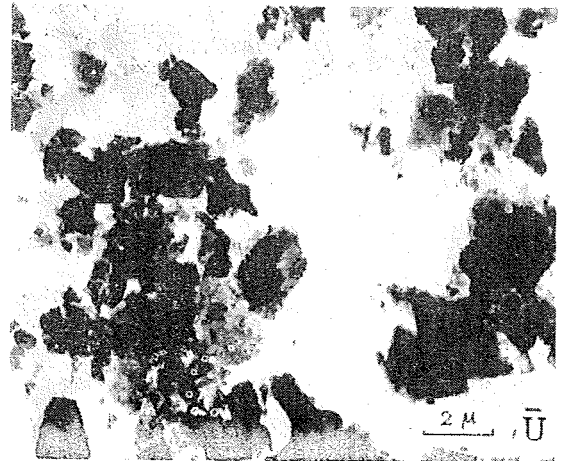
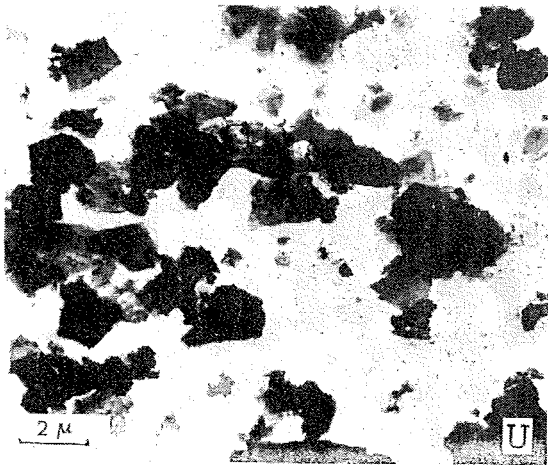
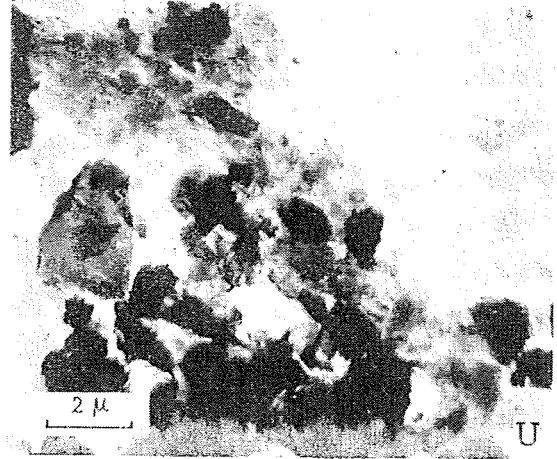
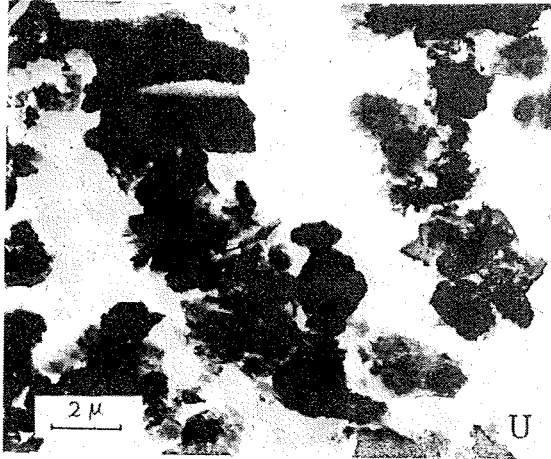


Plate III. Clay mineral (Kobayashi and Oinuma)

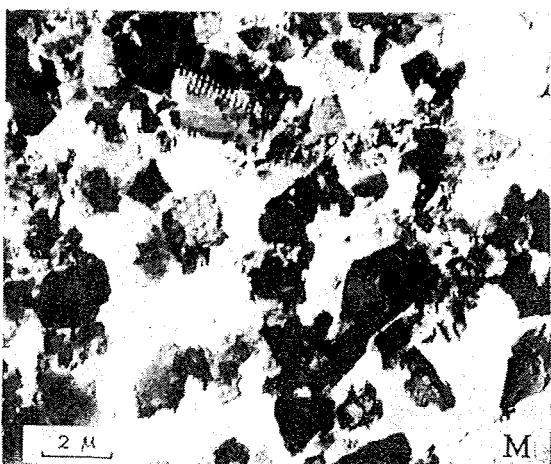
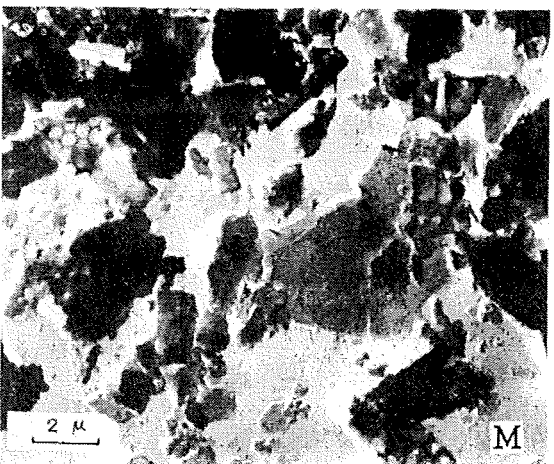
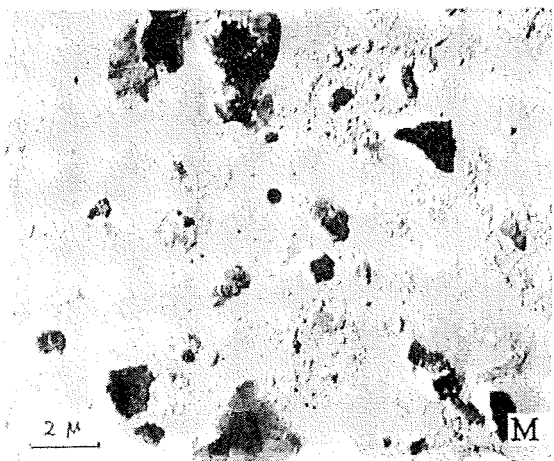
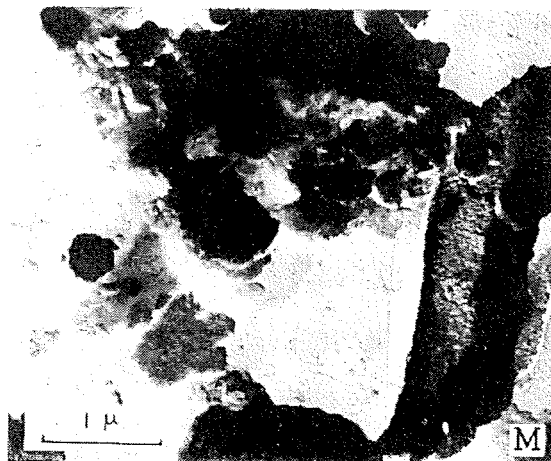
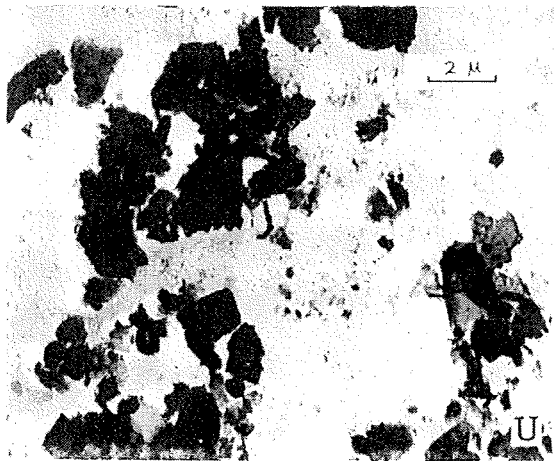
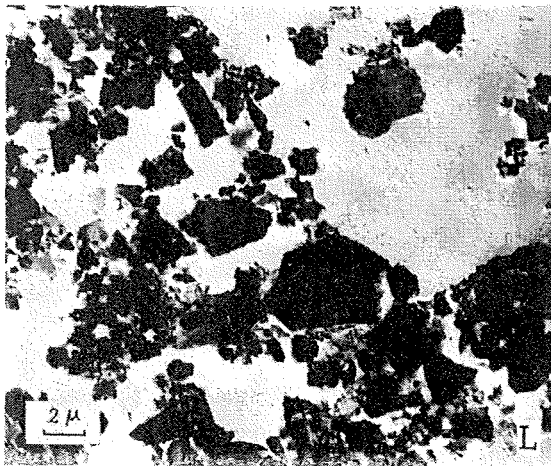
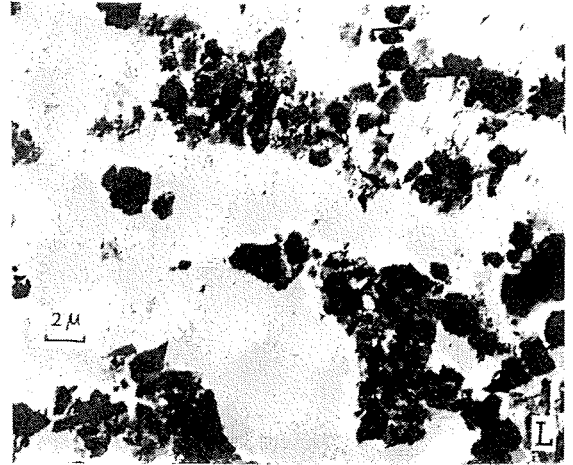
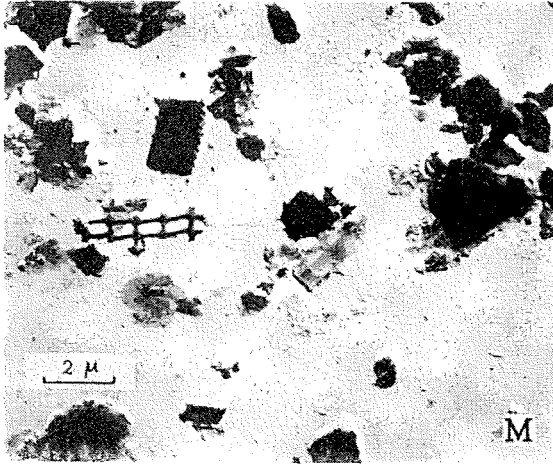


Plate IV. Clay mineral (Kobayashi and Oinuma)



U: The upper part specimen of the core sample.

M: The middle part specimen of the core sample.

L: The lower part specimen of the core sample.

K: Kaolinite from Hara district, Gifu Prefecture.

Q shows quartz line.

5. X-ray diffraction patterns of well oriented powders of clay fraction less than two microns.

(U): The upper part specimen of the core sample.

(M): The middle part specimen of the core sample.

(L): The lower part specimen of the core sample.

Plate II. Electron micrographs.

U: The upper part specimen of the core sample.

Plate III. Electron micrographs.

U: The upper part specimen of the core sample.

M: The middle part specimen of the core sample.

Plate IV. Electron micrographs.

M: The middle part specimen of the core sample.

L: The lower part specimen of the core sample.