INFRARED STUDY OF 10 Å TYPE GARNIERITE ON HEATING

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The 10 Å type garnierite from Nakey, New Caledonia was examined by means of thermal analysis and continuous high-temperature X-ray diffraction technique, and was heated at various temperature up to 1000 °C and after each treatment the specimens were cooled and also examined by X-ray powder diffraction and infrared analysis. The first basal reflection from this 10 Å type garnierite is a broad reflection, and its peak profile is asymmetric with a spreading towards smaller angles of 2 θ . The basal spacing exhibits little change when the specimens soaked in water or in ethylene glycol. The specimen shows the absorption bands at 3685 cm⁻¹, 3643 cm⁻¹ and 3625 cm⁻¹ in the OH region. The bands at 3690 cm⁻¹, 3670 cm⁻¹, 3645 cm⁻¹ and 3625 cm⁻¹ become clear in the temperature range from 250°C to 500°C. The band at 3690 cm⁻¹ becomes unrecognizable and the very sharp bands at 3675 cm⁻¹, 3660 cm⁻¹, 3645 cm⁻¹ and 3625 cm⁻¹ are recognized in the spectra of the specimens heated to 750°C and 800°C. These infrared absorption bands of this specimen which was heated between 750°C and 800°C, are in good agreement with the data of synthetic and natural nickel rich talc, except for strong intensity of the band at 3765 cm⁻¹. From the changes of these bands on heating, the material is considered to be either a mixture of nickel rich hydrated talc layer and magnesium rich hydrated talc layer or nickel rich hydrated talc layer with a heterogeneous distribution of Ni and Mg in octahedral layer, and contains extremely small amounts of chrysotile as impurity.

INTRODUCTION

Pecora *et al.* (1949) and Faust (1966) have recommended to use of garnierite as a general name for hydrous magnesium-nickel silicates. X-ray diffraction data have revealed that garnierites are commonly minerals with basal reflections of 7 Å, 10 Å, 12 Å and 14 Å. Many garnierites show both 7 Å and 10 Å basal spacings in X-ray powder diffraction patterns. It is generally accepted that the component having a 7 Å basal reflection resembles a serpentine group mineral. Nevertheless, the component having a 10 Å basal spacings is much uncertainly, and is frequently described as talc

and/or talc like layer, because this does not expand in water or in ethylene glycol. Kinoshita *et al.* (1960) showed the difference between talc and 10 Å type garnierite in the nature of dissolution in acid. Maksimovic (1966) concluded in his study on β -kerolite-pimelite series that a non expanding 10 Å layered pimelite predominate among the nickel clay minerals. Recently Brindley and Hang (1973) divided garnierites into 7 Å (serpentine-like) and 10 Å (talc-like) categories, based on the position of strong basal diffraction peak. They also suggested that the model structure of 10 Å garnierite is talc-like mineral with a high degree of layer stacking

disorder, and that the talc-like minerals with basal spacings near 10 Å are considered to be talc monohydrates $[(\text{Mg}, \text{Ni})_3\text{Si}_4\text{O}_{10}(\text{OH})_2] \cdot \text{H}_2\text{O}$.

In this paper, the infrared study of 10 Å type garnierite on heating is reported.

MATERIAL AND EXPERIMENTAL

The material used in this study was selected monomineralic samples suitable for detailed study from many garnierites. After X-ray examination, a specimen from Nakey, New Caledonia was selected to use in this study, because it shows dominantly 10 Å basal reflection. In hand specimen, garnierite from Nakey consists of bright apple-green parts and pale green parts on limonitized dunite. The material in bright apple-green parts showing 10 Å basal reflection was meticulously hand-picked in amounts sufficient for this study under a binocular microscope.

The material for X-ray, thermal and infrared analyses was prepared as follows:

The material was heated at an average rate of 5°C/min.. When heated to a required temperature, which was decided from the result of the thermal analysis, the material was rapidly cooled in a desiccator. In order to prevent rehydration, it was kept in nujol. The material for the infrared measurements of the region from 3000 cm^{-1} to 4000 cm^{-1} was prepared by the nujol paste method and that of region from 2000 cm^{-1} to 400 cm^{-1} was prepared by KBr tablet method.

The DTA and TGA curves were recorded simultaneously at an average heating rate of 5°C/min. with a Simultaneous Thermobalance Analyzer which was made by Rigaku Denki. The specimen was continuously heated in the furnace of X-ray diffraction

Unit at the heating rate of 5°C/min.. During the heating operation, diffraction pattern was recorded at 2θ intervals, containing the (001) reflection of the specimen. The infrared absorption spectra were obtained with a Hitachi EPI-G Grating Infrared Spectrophotometer.

THERMAL ANALYSIS

Simultaneous DTA and TGA curves for 22.4 mg of the present material are shown in Fig. 1. The initial rapid weight loss of 6.25%, corresponding to the loss of hygroscopic water, is largely completed by about 120°C. The second weight loss due to elimination of the remaining interlayer water is very slow, ranging from 120°C to 775°C, as indicated by the gentle slope of the TGA curve. In this step of weight loss, about 5.25% of the total weight is lost. The third weight loss of 2.50% due to dehydroxylation of constitution water starts at 775°C and terminates at 900°C. The DTA curve shows a large endotherm between room temperature and 110°C and a small endotherm between 800°C and 900°C.

Fig. 1. DTA and TGA curves of 10 Å type garnierite from Nakey, New Caledonia.

X-RAY ANALYSIS

The X-ray diffraction patterns of unheated and treated garnierite from Nakey

Fig. 2. X-ray diffraction patterns of 10 Å type garnierite with heating treatments and treated with ethylene glycol.

Note: 1: untreated
 2: heated to 500°C.
 3: heated to 750°C.
 4: heated to 900°C.
 5: heated to 1050°C.
 6: treated with ethylene glycol.

are shown in Fig. 2. The pattern of the natural specimen in Fig. 2 is similar to those given by Maksimovic (1966) and Brindley and Hang (1973) which they concluded β -kerolite or 10 Å type garnierite, and resembles to that given by Imai *et al.* (1973) which they named as hydrated talc. The first basal reflection from this specimen is an asymmetrical one with wing in the direction of small angle of 2θ . When the sample is heated up to 750°C, this

reflection is slightly sharpened as compared with those of the specimen which is untreated, treated with ethylene glycol and water. Enstatite is recognized in the X-ray diffraction pattern of the specimen heated at 900°C. At 1050°C, the specimen is transformed to mainly enstatite with minor olivine. Soaking the original sample in water and ethylene glycol has little effect on its 001 reflection.

The changes in d-spacings and intensity of the 001 reflection of the present specimen were observed by continuous high-temperature X-ray diffraction technique. The results are presented diagrammatically in Fig. 3. The 9.9 Å reflection is kept in d-spacings up to 200°C. Above this temperature range, this reflection is shifted to 9.66 Å. The intensity of this reflection is gradually and slightly increases up to 200°C, and decreases slightly in the temperature range between 300°C and 400°C. This reflection becomes to decrease from 700°C and then disappears at about 960°C.

Fig. 3. Changes in spacings and intensity of the basal reflection observed on heating.

CHEMICAL ANALYSIS

Chemical analysis and structural formulae of this mineral are given in Table 1. Structural formula under heading A in Table 1 is calculation on the basis of 4 Si, corresponding to talc formula $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$.

Table 1 Chemical analysis and structural formulae of 10 Å-type garnierite,

	# G - A Nakey New Caledonia
SiO ₂	49.06 %
Al ₂ O ₃	1.03
Fe ₂ O ₃	0.60
MgO	13.32
CaO	0.00
NiO	24.04
Na ₂ O	0.12
K ₂ O	0.03
H ₂ O (+)	7.48
H ₂ O (-)	4.26
Total	99.94

	A	B
Si	4.00	3.86
Al	0.10	0.10
Fe ³⁺	0.04	0.04
Mg	1.62	1.56
Ni	1.57	1.52
Na	0.19	0.19
K	0.03	0.03
Σ Oct.*	3.40	3.14
H ₂ O (+)	2.03	1.96

* Σ Oct. = Σ (R²⁺ + 3/2 R³⁺).

The composition of this mineral corresponds to the formula (Al_{0.10}Fe_{0.04}Mg_{1.62}Ni_{1.57})Si_{4.00}O_{11.40}·2.03 H₂O. The ratio of octahedral to tetrahedral cations is O/T=0.84, which lies between the ideal O/T ratios of 1.5 for serpentine-type mineral and 0.75 for a talc type mineral. Using the formulation proposed by Brindley and Hang (1973), based on the mole ratios of octahedral and tetrahedral cations, the composition corresponds to 77% talc type and 23% serpentine type. Under heading B, structural formula is calculated on the basis of a combined tetrahedral+octahedral cation valence of 22; that is total oxygen anion requires 11. In both formulae, Mg, Ni, Al and Fe³⁺ are considered to occupy octahedral positions and Σ (Octahedral cations) is taken as Σ (R²⁺+3/2 R³⁺).

INFRARED ANALYSIS

The untreated material gives the infrared absorption band at 3685 cm⁻¹ in addition to broad band at 3625 cm⁻¹ with a

Fig. 4. Infrared absorption spectra of 10 Å type garnierite, unheated and after heating at the temperatures indicated. Peak positions are given in wave number (cm⁻¹).

shoulder at about 3643 cm^{-1} and a broad band 3365 cm^{-1} in the OH region. The OH bending vibration is recognized at 1630 cm^{-1} . The garnierite contains one very large band near 1015 cm^{-1} and two bands at 668 cm^{-1} and 455 cm^{-1} , and other small bands such as 708 cm^{-1} , 600 cm^{-1} and 420 cm^{-1} .

The changes of the infrared absorption bands of this mineral on heating are shown in Fig. 4. The bands at 3685 cm^{-1} , 3665 cm^{-1} , 3645 cm^{-1} and 3625 cm^{-1} in the OH region become clear in the range from 250°C and 500°C . Four OH stretching vibrations in the region from 3600 cm^{-1} to 3700 cm^{-1} become very sharp in the temperature range from 750°C and 800°C , and yields further valuable information on the grouping of cations around the hydroxyl group. These bands persist up to 900°C . In the region between 1300 cm^{-1} and 400 cm^{-1} , the spectra of heated specimens become to change at about 850°C , that is; new bands appear at 942 cm^{-1} , 887 cm^{-1} , 862 cm^{-1} and 645 cm^{-1} on the infrared spectra. After heating to 900°C , the initial bands of untreated specimen can not be recognized on the infrared spectra of treated specimens. Infrared absorption bands of both forsterite and enstatite are recognized on the spectra of the specimen heated to 1000°C .

DISCUSSION

The changes in clay minerals in thermal treatment are made clear by X-ray, thermal and infrared analyses. Based on variations of infrared absorption spectra on heating various clay minerals, especially chlorite, vermiculite, sepiolite and palygorskite, Hayashi and Oinuma (1963) and Oinuma and Hayashi (1966) and Hayashi

et al. (1969) have found directly the behaviour of the OH groups on heating. They assigned the infrared absorption bands in the hydroxyl stretching region on the basis of behaviour during dehydration and/or rehydration of the above mentioned clay minerals.

Before further discussion of the matter, the behaviours of the infrared absorption bands in the OH region for magnesian clay minerals on heating are given in Fig. 5 for the sake of comparison.

Fig. 5. Schematic explanation of the infrared absorption bands in the OH region for magnesian clay minerals in the temperature range between 700°C and 850°C .

Talc has an absorption band at 3682 cm^{-1} before heat treatment. This band remains to be unchangeable after heating up

to 950°C, and then decreases in intensity on account of the endothermic reaction near 1050°C. After heating at 1100°C, this band is almost unrecognizable. This band is due to the OH stretching vibration. The degree of substitution of another divalent ions for Mg in talc splits into as many as four peaks in the region between 3600 cm^{-1} and 3700 cm^{-1} .

Hydrated talc gives a strong and sharp absorption band at 3675 cm^{-1} with a shoulder at 3660 cm^{-1} , and weak and broad bands at 3620 cm^{-1} and 3440 cm^{-1} . The bands at 3670 cm^{-1} and 3660 cm^{-1} are recognized on the spectrum of the specimen heated to 700°C. These bands are unrecognizable above the temperature for endothermic reaction near 825°C. These bands may be attributed to the OH stretching vibration in talc layer.

Mg-vermiculite has an endothermic reaction due to dehydration of interlayer water around 200°C, whereas in the infrared spectrum a broad absorption band is found centering 3380 cm^{-1} which is due to the rapid rehydration of vermiculite during the cooling process after heat treatment. After heated at 200°C and 400°C, an absorption band is observed at 3680 cm^{-1} and in further heating at 775°C and 800°C this absorption band becomes more distinct. However at the temperature higher than the endothermic and exothermic reactions near 830°C, this band disappears. Judging from the behaviour of the infrared absorption band at 3680 cm^{-1} in the heat treatment of Mg-chlorite and talc, the absorption band of vermiculite at 3680 cm^{-1} is supposedly due to OH in talc layer of vermiculite.

Mg-chlorite has two large bands at 3586 cm^{-1} and 3420 cm^{-1} , with a shoulder at 3680 cm^{-1} . Only the band at 3680 cm^{-1} ,

which is not very distinct so far, still remains when the specimen was heated at 750°C and 820°C. The disappearance of the two large OH bands at 3586 cm^{-1} and 3420 cm^{-1} after endothermic reaction at 630°C indicates these bands are assigned to the OH stretching vibration in brucite layer of chlorite structure. The endothermic reaction near 820°C is due to the loss of OH in talc like layer in the chlorite structure. The band at 3680 cm^{-1} becomes unrecognizable when the sample was heated above the endothermic reaction. From this fact, the band at 3680 cm^{-1} is attributed to OH stretching vibration in talc like layer of chlorite structure.

Stevensite gives a sharp absorption band at 3685 cm^{-1} and a broad band at 3430 cm^{-1} with a shoulder at 3625 cm^{-1} . After heating to 700°C, the sharpening of the band at 3675 cm^{-1} is conspicuous shifting slightly towards the lower frequency side. This band disappears in the dehydroxylated products heated to 1000°C.

Sepiolite gives rise to a sharp band at 3685 cm^{-1} , strong bands at 3617 cm^{-1} with a shoulder at 3645 cm^{-1} and at 3571 cm^{-1} , and broad bands at 3350 cm^{-1} and 3200 cm^{-1} . The sharp band at 3685 cm^{-1} shifts to near 3670 cm^{-1} by heat treatment at 610°C and 730°C, but this band disappears at 860°C which is the temperature of endothermic reaction. This band is assigned to the OH stretching vibration in octahedral layer of sepiolite structure.

Chrysotile gives rise to a very strong band at 3685 cm^{-1} having a shoulder at 3650 cm^{-1} . Although the band is gradually weakened with increase of the heating temperature, it remains at 750°C. Chrysotile has a very large endothermic peak between 600°C and 690°C, and a large exothermic peak at about 800°C. The

band at 3685 cm^{-1} is due to the OH stretching vibration. The band at 3645 cm^{-1} is attributed to the splitting effect which is observed in talc and amphiboles. The OH stretching vibrations of chrysotile persists until 750°C .

In the temperature range between 700°C and 850°C , the above mentioned minerals having the hydroxyl stretching vibrations are listed below.

Talc (950°C); probably including

Willemseite.

Hydrated talc (825°C).

Mg-vermiculite (830°C).

Stevensite (820°C).

Mg-chlorite (820°C).

Sepiolite (820°C).

Chrysotile (750°C).

Temperature of endothermic peak of each clay mineral is expressed numerals in the brackets.

In the temperature range between 850°C and 900°C , the hydroxyl stretching vibrations are seen only in talc among the above mentioned clay minerals.

The infrared spectra in the OH region of the present specimen which was heated between 750°C and 800°C , give sharp absorption bands at 3675 cm^{-1} , 3655 cm^{-1} , 3645 cm^{-1} and 3625 cm^{-1} . These bands are in good agreement with the published work on synthetic and natural nickel rich talc, except for strong intensity of the band at 3675 cm^{-1} (Wilkins and Ito, 1967; De Waal, 1970). Furthermore, the intensity ratios of the bands for the present specimen are unchangeable until 850°C . After heating of this sample to 900°C , the band at 3675 cm^{-1} decreases in intensity suddenly and the bands at 3660 cm^{-1} , 3640 cm^{-1} and 3620 cm^{-1} are observed. This fact also suggests that this specimen contains some magnesium bearing mineral

such as talc and hydrated talc.

It has been shown by Vedder (1964), Wilkins (1967) and Wilkins and Ito (1967), that the hydroxyl stretching fundamental peak for talc splits into as many as four peaks, depending on the degree of substitution of the magnesium in the octahedral layer by other divalent ions such as iron, nickel and cobalt. Vedder (1964) showed that relative intensities of these absorption bands could be expressed by $1 : 3x/1-x : 3(x/1-x)^2 : (x/1-x)^3$ where $(1-x)$ = Mg concentration, and x = the concentration of the ions replacing Mg in the octahedral site. This corresponds to a random distribution of the two species over M(1) and M(2) sites in the octahedral layers. From both formulation of this mineral recorded in Table 1, the Mg concentration $(1-x)$ in the octahedral layer of this specimen is obtained 0.47 and 0.49 respectively. When the Mg concentration $(1-x)$ and Ni concentration (x) in the octahedral layer of this specimen were assumed to be equal (0.5), the calculated intensity ratio of four splitting OH stretching bands was indicated $N_A:N_B:N_C:N_D = 1:3:3:1$. In practice, however, the measured intensity ratio of these bands in the infrared spectrum of the specimen heated to 750°C is $1:0.78:1.03:1.27$. The measured intensity ratio of the N_A and N_D band resembles the calculated values, but the intensity ratio among the N_A , N_B and N_C bands do not resemble those calculated values. Using the measured intensities of N_B , N_C and N_D in the infrared spectra of the heated specimens, Mg concentration was obtained as follows.

In the specimen heated to 750°C

Mg concentration calculated from N_B :

$N_C = 0.44$

Mg concentration calculated from N_C :

$N_D = 0.18$ average 0.310

In the specimen heated to 800°C

Mg concentration calculated from N_B :

$$N_C = 0.44$$

Mg concentration calculated from N_C :

$$N_D = 0.21 \quad \text{average } 0.325$$

When Mg concentration was assumed 0.32, the calculated intensity ratios of $N_A:N_B:N_C:N_D$ was revealed to 0.1:0.64:1.35:0.96. The calculated intensity of N_A is about one tenth of the observed intensity. This fact indicates experimentally and theoretically that a part of the strong intensity of the band at 3675 cm^{-1} (N_A) would be ascribed to the presence of the material consisting of either magnesium bearing minerals such as talc and hydrated talc or nickel rich hydrated talc layer with heterogenous distribution of Mg with Ni in octahedral layer.

The spectrum of the present specimen in the region between 2000 cm^{-1} and 400 cm^{-1} at room temperature closely resembles to that of nickel rich talc except for the presence of the band around 600 cm^{-1} . The band around 600 cm^{-1} disappeared in the temperature range between 500°C and 750°C. The band at 3685 cm^{-1} in the OH region showed same behaviour in this temperature range. From these thermal behaviours of these bands around 600 cm^{-1} and at 3685 cm^{-1} they might be attributed to chrysotile (Koshi *et al.*, 1969). Nevertheless, DTA curve of this specimen has neither a characteristic endothermic peak between 630°C and 690°C with weight loss nor a characteristic exothermic peak between 790°C and 810°C of chrysotile. Of course, X-ray powder diffraction pattern of this specimen has not a 7 Å reflection. However, extremely small amounts of chrysotile is found by the transmission electron microscope.

Of special interest are the intensity ratios of the two absorption bands at 668

cm^{-1} and 705 cm^{-1} . The intensity ratios of these bands give a good indication of the Mg and Ni contents of the minerals. The band at 705 cm^{-1} increases in its intensity until 850°C. The intensity of the two bands in the region of approximately 700 cm^{-1} for $\text{Mg}_{32}\text{Ni}_{68}$ talc like layers which is calculated from relative intensity ratio of $N_B:N_C$ and $N_C:N_D$ in the spectra of the specimens heated to 700°C and 800°C, could be presumed nearly equal intensities. Actually the intensity ratios of the bands at 668 cm^{-1} to the band at 705 cm^{-1} show about 1:2 in the spectra of the specimens heated to 750°C, 800°C and 850°C. The reason why the intensity of the band at 668 cm^{-1} is higher than that of the band at 705 cm^{-1} , would be attributed to the existence of magnesium rich talc besides nickel rich talc.

Conclusively, the present specimen is either a mixture of Ni rich hydrated talc layer and Mg rich hydrated talc layer or nickel rich hydrated talc layer with a heterogenous distribution of Ni and Mg in octahedral layer, and contains extremely small amount of chrysotile as impurity.

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加熱処理した 10 Å 型ガーニーライトの赤外線による研究

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ニューカレドニア産の 10 Å 型ガーニーライトを示差熱分析、熱重量分析、連続加熱高温 X 線分析により調べた。また試料を 1000°C までの種々の温度に加熱した後に、X 線分析と赤外線分析を行なった。未処理の 10 Å 型ガーニーライトの底面反射は巾広く、 2θ の角度の小さい方に翼を広げた非対称形をしている。この底面反射は水やエチレン・グリコール処理では変化しない。10 Å 型ガーニーライトの OH 領域の赤外線吸収スペクトルは 3685 cm^{-1} , 3643 cm^{-1} と 3625 cm^{-1} が認められるが、250°C～500°C 加熱処理を行なうと、 3690 cm^{-1} , 3670 cm^{-1} , 3645 cm^{-1} と 3625 cm^{-1} の吸収が明瞭になり始め、750°C と 800°C の加熱により、 3690 cm^{-1} の吸収が認められなくなり、 3675 cm^{-1} , 3660 cm^{-1} , 3645 cm^{-1} , と 3625 cm^{-1} の鋭い吸収が認められる。これらの吸収は 3675 cm^{-1} の吸収の強度が強いことを除くと、人工および天然産のニッケルに富むタルクの吸収とよく一致する。この鉱物の OH 領域の赤外線吸収スペクトルの変化および吸収の強度比の計算から、この鉱物はニッケルに富む加水タルクとマグネシウムに富む加水タルクの混合物であるか、または八面体層中でニッケルとマグネシウムの不規則な分布をもつニッケルに富む加水タルクであると考えられる。またこの試料には極く微量のクリソタイルが不純物として含まれている。