NESQUEHONITE FROM YOSHIKAWA, AICHI PREFECTURE, JAPAN : OCCURRENCE AND THERMAL BEHAVIOUR

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Nesquehonite, $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$, from Yoshikawa, Aichi Prefecture, the first occurrence in Japan is described. It is found as columnar aggregates covering the encrustment of "yoshikawaite" on the weathered surface of the serpentinite. The X-ray diffraction pattern and the optical properties are identical to those of the previous data.

The nesquehonite is thermally decomposed lastly to periclase through the three intermediate phases, that is, the dehydrate phase (80–200°C), the amorphous phase (210–270°C) and the decarbonate phase (280–480°C). Periclase is produced at 370°C. A few absorption bands characteristic of the decarbonate phase are already found in the infrared spectrum of the amorphous phase.

DTA curve for nesquehonite shows six endotherms with maxima at 132, 176, 216, 420, 507 and 526°C, respectively, and an exotherm at 499°C. The first three endotherms are attributed to the removal of H_2O , and the others to that of CO_2 . The exothermic reaction is considered to be coupled with the fifth endothermic one.

1. INTRODUCTION

Nesquehonite, $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$, one of the simple magnesium carbonate hydrate minerals, from Yoshikawa, Aichi Prefecture was found for the first time in Japan. The X-ray diffraction, DTA curves and optical properties are identified with those of the nesquehonite reported by other workers. It occurs closely associated with an unusual magnesium carbonate hydrate mineral, $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ *** which covers the serpentinite body as the encrustment, having a characteristically long spacing of 33.2 Å (Suzuki and Ito, 1973). Other natural occurrences of nesquehonite are, for example, (1) in cauliflower-like rough crust on the small wall of upper Juarssic dolostone cave of West Germany, admixed with hydro-

magnesite and trace of monohydrocalcite, calcite and aragonite (Fischbeck and Müller, 1971), (2) in serpentinite, (3) in coal mine (Winchell and Winchell, 1951) and so on. It was also found even in the carbonate scales in the air scrubber of an air-conditioning plant, associated with hydrocalcite and calcite (Marschner, 1969). A mixture of nesquehonite and aragonite was reported to be formed from the sea-water by the vapour phase diffusion technique (Towe and Malone, 1970).

Some mineralogical and crystallographical studies of nesquehonite have been reported, most of which were done for synthesized specimens. Stephan and Macgillavry (1972) determined the crystal structure of nesquehonite synthesized by the method of

*** Application for an approval of the name, "yoshikawaite," is pending.
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Menzel and Brückner. Since it has some noticeable behaviour indicating the change into periclase due to thermal decomposition, keen attention has been paid. It passes through the stages accompanied by a few intermediate phases (Morandi, 1969; Iwai *et al.*, 1969).

In this paper, the occurrence and the thermal behaviour of nesquehonite from Yoshikawa will be presented in detail based on the characteristics in X-ray diffraction patterns, DTA-TG curves and infrared spectra.

The optical properties of nesquehonite are as follows: $\alpha < 1.448$, $\beta = 1.504$, $\gamma = 1.528$; $\gamma - \alpha > 0.080$, $\gamma - \beta = 0.024$. It is colourless and shows nearly straight extinction with positive elongation.

2. MODE OF OCCURRENCE

Nesquehonite is nearly colourless and transparent, and slender column-like crystals (Fig. 1). It occurs as columnar aggregates growing perpendicular to the surface of the encrustment of "yoshikawaite" covering the serpentinite body (Fig. 2).

Fig. 1. Scanning electron microphotograph of nesquehonite.

The serpentinite body contains some amounts of brucite and is conspicuously carbonate-hydrated, forming hydrotalcite group-mineral, hydromagnesite and artinite. Aragonite and hydromagnesite coexist also as vein-fillings in the serpentinite. As reported in the previous paper (Suzuki and Ito, 1973), the encrustment of "yoshikawaite" is an evaporation product, on the surface of the cutting cliff, from the groundwater near the surface which dissolves brucite, hydrotalcite group-mineral and the related minerals. This unusual "yoshikawaite" is slightly soluble even in distilled water and is more soluble in rain water. Judging from the occurrences, nesquehonite is also considered to be an evaporation product on the surface of "yoshikawaite" mainly from rain water which dissolves the latter mineral. Brucite, hydrotalcite group-mineral, hydromagnesite and other magnesium carbonate hydrate minerals are soluble slightly in the water with dissolved CO_2 . Nesquehonite is observed to be formed by evaporation from such a solution at room temperature. However, in Yoshikawa nesquehonite

Fig. 2. Nesquehonite covering the encrustments of "yoshikawaite."

Table 1. X-ray powder diffraction data for nesquehonite from Yoshikawa, Aichi Prefecture, Japan.

Room temperature		60°C		100°C		440°C	
d	I/I ₀	d	I/I ₀	d	I/I ₀	d	I/I ₀
6.544	100	6.520	100	5.258 b	100	6.050	100
6.067	3	6.071	3	3.844	11	4.272	10
4.928	5	4.923	4	3.254 b	34	3.489	57
4.158	3	4.162	4	2.775 b	75	3.018	16
3.865	98	3.860	97	2.619	41	2.698	7
3.593	24	3.590	22	2.332	7	2.464	7
3.227	18	3.241	17	2.144	2	2.282	33
3.038	40	3.037	33	1.8607	27	2.134	72
2.987	10	2.986	9	1.7262	11	2.012	21
2.789	20	2.875	17			1.9073	13
2.627	18	2.625	20			1.8193	13
2.514	41	2.512	34	180°C		1.7423	5
2.328	5	2.341	5			1.6743	10
2.318	3	2.315	2			1.5564	8
2.176	14	2.203	3	5.203 b	100	1.5062	28
2.127	3	2.175	11	3.243 b	62	1.4629	13
2.023	13	2.126	3	2.804 b	54	1.4202	7
1.9294	34	2.222	10	2.764 b	92	1.3847	7
1.8953	3	1.9275	28	2.621 b	46	1.3171	5
1.8483	6	1.8943	2	1.858 b	31		
1.8382	6	1.8466	5				
1.8025	12	1.8385	5				
1.7764	2	1.8009	10	350°C		500°C	
1.7277	2	1.7751	3				
1.7208	3	1.7265	4				
1.6510	7	1.7181	4	6.042	100	2.442	9
1.6345	3	1.6587	2	4.266	10	2.118	100
1.6211	3	1.6496	7	3.488	66	1.4986	53
1.5861	2	1.6337	3	3.019	20	1.2765	6
1.5545	4	1.6211	2	2.702	11		
1.5295	2	1.5881	2	2.462	14		
1.5073	2	1.5535	4	2.283	43	(520°C*)	
1.4584	2	1.5291	2	2.135	50		
1.4342	2	1.5082	2	2.012	18		
1.4000	2	1.4588	1	1.9095	14	4.806	23
1.3854	2	1.4340	2	1.8190	11	2.449	12
1.3204	2			1.7420	7	2.373	37
1.3123	2			1.6729	10	2.118	100
1.2847	2			1.5576	10	1.7979	14
1.2588	2			1.5084	10	1.5721	14
1.2492	2			1.4633	11	1.4957	58
				1.4219	11		
				1.3816	10		
				1.3502	7		

* Left alone at room temperature for three months after heated to 520°C.

Fig. 3. X-ray diffraction patterns for nesquehonite and its thermal decomposition products. CuK_α radiation was used for 2θ .

occurs in association with only "yoshikawaite" and not with brucite and any other carbonate hydrate minerals.

3. X-RAY DATA

CuK_α radiation was used. The thermal effects on the whole X-ray diffraction patterns of nesquehonite are shown in Table 1 and Fig. 3. It is heated in air for about 10 to 20 minutes at each 10 or 20°C interval between 40 and 520°C on the Pt-heating stage of the high temperature X-ray diffraction apparatus. All measurements were made for the specimens cooled to room temperature after heating. Four decomposed phases are recognized with rising temperature. The first is the dehydrate phase which appears at 80°C. At about 100°C it

has the strongest reflection peak for 16.9° (2θ). Above this temperature the peak decreases in height gradually and completely disappears at least at 230°C, showing the formation of amorphous phase (the second phase). The dehydrate phase has generally broad peaks, whose reflection lines are mostly inherited from those of nesquehonite, and the lattice is slightly contracted with rising temperature. The third is the decarbonate phase and will be discussed in later section. It appears at 280°C showing very sharp lines, and disappears suddenly at 490°C. Periclase, the fourth phase, begins to appear at 370°C and increases gradually in crystallinity with rising temperature, followed by slight contraction of the lattice. However, magnesite is not recognized throughout the decomposition process.

The periclase formed from nesquehonite by heating up to 520°C was left alone in air at room temperature for about three months, whose diffraction datum is given in the last column of Table 1. A few new reflection lines appear together with those of periclase. The reproduced phase shows the d-spacing similar to that of brucite.

4. THERMAL ANALYSIS

Simultaneous DTA and TG curves of nesquehonite are shown in Fig. 4, together with that of the periclase from the 520°C-heated nesquehonite, mentioned in the above section. The heating rate is 10°C per minute in air. There are six endothermic reactions, 132, 176, 216, 420, 507 and 526°C, and an exothermic reaction at 499°C. The endotherm at 486°C is considered to be due to the coupling of the endothermic reaction at 507°C and the very rapid exothermic reaction at 499°C.

TG curve shows the continuous weight

Fig. 4. DTA-TG patterns: A, nesquehonite; B, periclase from the 520°C-heated nesquehonite. The heating rate is 10°C/min.

Table 2. The thermal decomposition products of nesquehonite at various temperatures between room temperature and 1000°C in air. The heating rate 10°C per minute.

Temperature (°C) in DTA curve	Decomposition products	Lost component (mole)
1. Room Temp.	$\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$	
2. 160	$\text{MgCO}_3 \cdot 1.57\text{H}_2\text{O}$	$1.43\text{H}_2\text{O}$
3. 209	$\text{MgCO}_3 \cdot 0.77\text{H}_2\text{O}$	$0.80\text{H}_2\text{O}$
4. 351	MgCO_3 1)	$0.77\text{H}_2\text{O}$
5. 457	$\text{MgO} \cdot 0.63\text{CO}_2$ 2)	0.37CO_2
6. 499	$\text{MgO} \cdot 0.44\text{CO}_2$	0.19CO_2
7. 511	$\text{MgO} \cdot 0.27\text{CO}_2$ 3)	0.17CO_2
8. 549	$\text{MgO} \cdot 0.03\text{CO}_2$ 3)	0.24CO_2
9. 1000	MgO 3)	0.03CO_2

Total weight loss in TG curve: 70.5%

1) amorphous phase.

2) decahydrate phase and periclase in X-ray pattern.

3) periclase in X-ray pattern.

loss until the last reaction is completed. Judging from the first appearance of periclase in the X-ray pattern at 370°C, the three endotherms at lower temperature region are related to the escape of H_2O , and the other three at higher temperature region to the escape of CO_2 . If the weight loss for each endothermic reaction is estimated from the TG curve for each temperature range, shown on the DTA curve of Fig. 4, the variations in the chemical compositions of the residuals through each reaction with rising temperature may be suggested as shown in Table 2. There is very slight weight loss towards higher temperature even after the last endothermic reaction is finished at about 550°C. It attains about 0.03 mole ratio of CO_2 at 1000°C. This weight loss may be regarded as an observational error. However, it is significant to note that the periclase from nesquehonite heated even up to 1000°C is also chemically active and is found to adsorb some amounts of H_2O and CO_2 at room temperature. The periclase from the 520°C-heated nesquehonite, left alone in air at room temperature for about three months, shows a characteristic DTA-TG curve as shown in Fig. 4. There occur two endotherms at 102 and 385°C, respectively. The former reaction is caused by the escape of H_2O and the latter by the escape of CO_2 . The total weight loss is 28.6%, made up as follows: 7.8% of H_2O and 20.8% of CO_2 , respectively.

A few DTA curves by the various heating rates have been reported. The pattern in this paper is rather similar to that by Morandi (1969), except a few differences. He showed the endotherms at 160, 190, 220, 430 and 525°C, and the exotherm at 515°C. The first two endotherms are higher than those in this paper. The exotherm in his

paper is conspicuously strong unlike those reported by the other workers. All endotherms below 300°C are generally attributed to the escape of H_2O . Beck (1950) and Morandi (1969) assigned the endotherm at about 430°C to the escape of OH. However, judging from the detailed investigation of the TG curve, it may be safe to attribute the endotherm to the escape of CO_2 . This is also supported by the appearance of periclase on X-ray diffraction patterns even at 370°C. Therefore, the third phase is considered to be the decarbonate. The endotherm around 520°C has been attributed to the escape of CO_2 . The nesquehonite from Yoshikawa shows two coupled endotherms at 507 and 526°C, respectively, both of which are from the escape of CO_2 . But the former reaction is very complicated, as it is coupled with the exothermic one. After all, nesquehonite from this area is thermally decomposed to periclase through the three steps of the escape of H_2O and those of CO_2 , respectively.

5. INFRARED ABSORPTION SPECTRA

The infrared spectra of nesquehonite and its heated samples are shown in Fig. 5 and the frequencies of the absorption bands in Table 3. The heated ones are the same as those used for the X-ray diffraction. All measurements were made at room temperature by the KBr disc technique. The two extra bands at 3670 and 3620 cm^{-1} indicated by asterisks for non-treated nesquehonite are due to the admixed chrysotile and "yoshikawaite," respectively. Since purified samples were used for heating experiment, the two extra bands do not appear on the other spectra.

Characteristic changes in the thermal decomposition will be described for each

range of the frequencies.

(A) 4000 to 2000 cm^{-1}

A sharp and strong absorption band at 3540 cm^{-1} for the non-treated nesquehonite is due to the vibration of O-H stretching and disappears at 60°C. A sharp band at 3550 cm^{-1} is observed at 60°C and as a shoulder at 100°C. Very sharp bands at 3680 cm^{-1} are found for the samples heated at 440 and 500°C. The absorption is clearly caused by the water adsorbed by the periclase formed from nesquehonite. A sharp

Fig. 5. Infrared absorption spectra of nesquehonite and the minerals heated at various temperatures.

Table 3. Infrared absorption data for nesquehonite from Yoshikawa, Aichi Prefecture.

Room Temp.	60°C	100°C	180°C	250°C	290°C	350°C	440°C	500°C
3670*	w	3550 m	3550 vw	3410 b s	3410 b s	3675 vw	3675 vw	3680 s
3620**	w	3430 vw	3395 b s	1505 b w	2630 b w	3410 b s	3415 b m	3405 b s
3540	w	3325 b s	3110 b w	1420 b s	2380 vw	2640 b vw	2930 vw	2930 w
3410	s	3150 vw	2505 vw	1104 b w	2340 w	2380 vw	2850 vw	2850 w
3130	vw	2625 vw	1508 b s	1010 w	1424 b s	2350 m	2630 b vw	2380 vw
2610	vw	2515 vw	1403 b s	851 m	1094 b w	1422 b s	2380 vw	2350 w
2500	vw	1640 w	1104 m	705 b w	1014 w	1094 b w	2350 m	1498 b w
1639	vw	1587 vw	1010 vw		854 m	1014 w	1430 b s	1422 b s
1555	vw	1560 vw	980 b m		705 b w	856 m	1098 b w	1130 vw
1540	vw	1543 vw	880 vw		660 w	709 b w	1014 w	1079 b w
1513	s	1520 s	846 m			661 w	853 vw	1016 w
1474	s	1468 vw	693 m				844 m	845 m
1418	s	1422 s	630 b m				710 b w	
1117	w	1099 w					660 w	
1095	w	1014 w						
1013	w	854 m						
948 b	m	704 w						
878	w	657 b w						
849	m	610 b w						
797 b	w	494 vw						
707 b	w							
595	m							
481	w							

* Chrysotile, ** "Yoshikawaite"

band at 2350 cm^{-1} and the shoulder at 2380 cm^{-1} appear at least at 250°C , increasing in intensity above this temperature, and disappear below 500°C . Since this temperature range is assigned to the amorphous phase and the decarbonate phase, the two absorption bands are derived from newly formed bonding in the decarbonate phase. It is noted that this new bonding is already formed in the amorphous phase at 250°C . Furthermore, an indication of this bonding is seen even at 180°C as a weak and broad band. The intensity of this band increases at 290 to 350°C , though the frequency is somewhat less by about 15 cm^{-1} . Two sharp but weak absorptions at 2930 and 2850 cm^{-1} may probably belong to the decarbonate phase, since no absorption is found in the same frequency region for the sample heated at 500°C . Or, they might be attri-

buted to the water adsorbed into KBr from the decarbonate phase rather than from periclase, although this phase does not show any signs of adsorption of water in the X-ray diffraction pattern.

(B) 2000 to 1300 cm^{-1}

Four distinct absorption bands at 1639, 1513, 1474 and 1418 cm^{-1} are observed at room temperature. All bands become diffused with rising temperature. A band by H-O-H bending appears at 1639 cm^{-1} and disappears at least at 100°C . The band at 1474 cm^{-1} is not distinctly observed at 60°C . The other absorption bands at 1513 and 1418 cm^{-1} become broad with rising temperature. Newly formed bands are observed at 1587 cm^{-1} at 60°C and 1385 cm^{-1} at 500°C . The latter is a diagnostic one for the periclase from the heated nesquehonite, in which a large amount of H_2O and CO_2

is adsorbed.

White (1971) showed three bands at 1518, 1470 and 1415 cm^{-1} to be caused by the absorption of CO_3^{2-} stretching. However, it is interesting to note that all bands become gradually diffused with rising temperature, in spite of the fact that the decarbonate phase shows higher crystallinity in X-ray diffraction patterns.

(C) 1300 to 800 cm^{-1}

The dehydrate phase, the decarbonate phase and the periclase from the heated nesquehonite are characterized by the broad bands at 980, 1094 and 1075 cm^{-1} , respectively. The absorption at 1095 cm^{-1} probably by CO_3^{2-} stretching in thermally non-treated nesquehonite is stronger at 100°C than those below this temperature, and then become broad with rising temperature. The bands at 948 and 878 cm^{-1} disappear completely at 60 and 180°C, respectively. The latter band may correspond to that of "yoshikawaite." However, it should be attributed to nesquehonite, because the "yoshikawaite" heated to 180°C shows the bands as strong as that at room temperature. The sharp band at 849 cm^{-1} by CO_3^{2-} bending becomes broad with rising temperature up to 440°C. Another characteristic band of the periclase from the heated nesquehonite is observed at 860 cm^{-1} .

(D) 800 to 400 cm^{-1}

The absorption spectra in this frequency region change rapidly with rising temperature. All bands become broad even at 60°C. The dehydrate phase and the decarbonate phase are characterized by the bands at 630 and 660 cm^{-1} , respectively. The periclase from the heated nesquehonite does not show any distinguishable absorption spectrum. The band at 797 cm^{-1} is also compatible with that of "yoshikawaite." But, judging from the same reason as mentioned above, it is also ascribed to the absorption by nesquehonite.

6. DISCUSSIONS

Some intermediate products through the thermal decomposition of nesquehonite have been reported by many workers. But all of these are not always crystalline by X-ray diffraction. As mentioned above, three crystalline phases and an amorphous one are identified. A schematic representation of the thermal decomposition of this mineral may be compiled as in Fig. 6.

The thermal decomposition of the sample heated for the X-ray diffraction is considered to proceed rapidly enough so as to form the metastable phases within ten to twenty minutes at various temperatures,

Fig. 6. Schematic diagram of the thermal decomposition of nesquehonite. v & Δ: endothermic and exothermic maxima, respectively. o: temperature on DTA curve. ESQ.: nesquehonite.

since even the sample held for more than 24 hours shows no significant change in the X-ray pattern. The samples for DTA-TG weigh about 15 to 20 mg, but those for the X-ray analyses about 1 g. In both thermal treatments, the heating rates are approximately the same. Therefore, the "holding" effect on the larger amount of sample is considered to correspond to the continuous uprising of temperature for the less amount of sample for DTA-TG with the constant rate of 10°C per minute.

The chemical composition of the dehydrate phase changes gradually from $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ to $\text{MgCO}_3 \cdot 0.77\text{H}_2\text{O}$ with rising temperature. However, three distinguishable endotherms with continuous weight loss are found on the DTA curve. Judging from the X-ray patterns, this phase is related to the first two endotherms. As all the reflection lines become broad above 120°C, the thermal condition below this temperature corresponds probably to the endothermic reaction of the maximum at 132°C. Then, the broad peaks for each pattern gradually disappear with rising temperature through the endotherm at 176°C, and the patterns of the amorphous phase appears.

The amorphous phase contains some amounts of H_2O , that is, 0.77 mole which is equal to 25.7% of total H_2O . It is continuously lost probably up to 351°C, through the endotherm at 216°C. However the decarbonate phase appears already at 280°C in the X-ray pattern. As strikingly shown in the infrared spectra, the absorption bands characterized by this phase are already observed even in the amorphous phase at 250°C. Therefore, the formation of new chemical bonds suggestive of the decarbonate phase is distinctly proceeding in the amorphous phase. The decarbonate

phase between 410 and 440°C shows the strongest peak heights of the X-ray reflection lines, though the subsequent phase, periclase, is already formed in some amounts. The decrease in intensity of the absorption at 2350 cm^{-1} in the sample heated to 440°C is caused by the decrease in quantity due to its decomposition. It is completely decomposed by heating from 480 to 490°C without any diffused X-ray patterns. This temperature range corresponds to the exothermic reaction of the maximum at 499°C. Therefore, the decomposition is completed just before this exothermic reaction starts. The chemical composition of this phase is about $\text{MgO} \cdot 0.44\text{ CO}_2$ at 499°C. Only periclase is observed in the X-ray diffraction patterns above 490°C, which show, however, broad reflection lines. It is interesting to note that some amounts of CO_2 are contained in the periclase, and is released gradually with rising temperature up to 550°C. Judging from the two distinct endotherms at 507 and 526°C, the CO_2 may be loosely joined to very fine-grained and less crystalline periclase by two somewhat different ways.

Explanations of the exothermic reaction have been discussed by many workers. The magnesium carbonate hydrate minerals are generally characterized by this exotherm and the subsequent endotherm. It is reported that this exotherm might be caused by the crystallization of periclase with a question mark added in the table (Beck, 1950). Morandi (1969) concluded that it is attributed to the formation of magnesite, based on the DTA data under CO_2 condition. On the other hand, Iwai *et al.* (1969) suggested the abrupt contraction of the lattice of periclase through the process of increasing in crystallinity around 500°C. As

already mentioned above, magnesite is not recognized in air, and the lattice of periclase also contracts gradually with rising temperature. However, this exothermic reaction is drastic and even explosive, especially in hydromagnesite and "yoshikawaite." It might have been caused by some other reactions which can release a great amount of energy within a short time. Further study on this topic will be reported in the near future.

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愛知県吉川産 nesquehonite

鈴木 重人, 伊藤 正裕

Nesquehonite ($\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$) が、含ブルーサイト蛇紋岩の風化面を覆う "yoshikawaite" の表面から発見され、X線回折および光学的性質に基づき同定された。無色透明の柱状結晶をなす。本鉱物は、温度の上昇とともに、脱水相、非晶質相および脱炭酸相を経てペリクレスへ分解する。非晶質相の IR パターンには、すでに脱炭酸相を特徴づける吸収帯がある。また、DTA では 132, 176, 216, 420, 507, 526°C に吸熱、499°C に発熱を示す。この発熱反応は、含水マグネシウム炭酸塩鉱物群に共通しており、この原因について言及した。