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Li-bearing Tosudite from the Sungsan Mine, Korea

해남 성산광산에서 산출되는 함리튬 토수다이트

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ABSTRACT: Tosudite from the Sungsan mine, Korea is an alteration product of rhyolitic tuff in the Cretaceous Hwangsan Formation. It is associated with illite, dickite, nacrite or quartz and also found in the cavities of black claystone. X-ray diffraction and chemical analyses show that the Sungsan tosudite is a lithium-bearing aluminous 1:1 regularly interstratified mineral of di, dioctahedral chlorite and smectite. Its structural formula is $(K_{0.73}\,Na_{0.02}\,Ca_{0.07})\,(Si_{13.23}\,Al_{2.77})\,(Li_{0.52}\,Mg_{0.08}\,Mn_{0.01}\,Fe^{3+}_{0.07}\,Al_{12.33})O_{40}(OH)_{20}$ and it suggests that Sungsan tosudite consists of regularly interstratified Li-donbassite and beidellite. DTA and TG curves as well as IR absorption data also support such a result. Temperature of formation of tosudite is inferred to be between 110℃ and 270℃.

요약: 토수다이트(tosudite)는 녹니석/스멕타이트의 규칙적 혼합층광물(regularly interstratified mineral of chlorite/smectite)로서, 성산광산 주변의 열수변질작용을 받은 백악기 황산응회암층 내에서 발견된다. 이 광물은 주로 일라이트, 딕카이트, 나크라이트 및 석영에 수반되어 산출되며, 때로는 흑색의 납석 내 공극을 나크라이트 또는 석영과 함께 채우기도 한다. X-선회절분석, 화학분석, 시차열분석, 열중량분석 및 적외선분광분석에 의하여 성산 토수다이트는 dioctahedral chlorite와 dioctahedral smectite가 1:1로 규칙적으로 혼합층을 이룬 광물임이 확인되었다.

EPMA분석과 ICP-MS분석에 의하면 성산 토수다이트는 많은 양의 알루미늄을 포함하고 있으며, 철과 마그네슘의 양은 적다. 또한 상당한 양의 리튬(0.42 wt. %)을 함유하고 있다. 성산 토수다이트의 구조식은 산소 50을 기준으로 할 경우 다음과 같다:
$(K_{0.73}Na_{0.02}Ca_{0.07})\,(Si_{13.23}Al_{2.77})\,(Li_{0.52}Mg_{0.08}Mn_{0.01}Fe^{3+}_{0.07}Al_{12.33})O_{40}(OH)_{20}$
화학분석치로부터 성산 토수다이트는 리튬을 함유하는 돈바사이트(donbassite)와 층간이온이 칼륨인 바이델라이트(beidellite)의 혼합층광물임을 알 수 있다. 이와 같은 결과는 열분석과 적외선흡수분광분석 자료에서도 확인된다.

성산광산 주변의 열수분대에 의하면, 성산 토수다이트의 생성 온도는 초기 딕카이트화작용과 후기 알바이트화작용의 중간에 해당하는 110°~270℃로 추정된다.

INTRODUCTION

The name tosudite was given by Frank-Kamenetskii et al. (1965) to a 1:1 regularly interstratified dioctahedral chlorite/smectite after Professor Dr. Toshio Sudo, the first researcher of the mineral (Sudo et al., 1954). Since then tosudite has been usually used for a regularly interstratified mineral of di, dioctahedral or di, trioctahedral chlorite component. Tosudite has been reported from Japan by many different authors (Mitsuda, 1957; Hayashi, 1961; Kanaoka, 1968;

Shimoda, 1969; Ichikawa and Shimoda, 1976; Sudo and Shimoda, 1978; Inoue and Utada,1989). In addition Li-or Cr-bearing tosudite also has been reported (Brown et al., 1974; Nishiyama et al., 1975; Maksimovic and Brindley, 1980; Creach et al., 1986; Foord et al., 1986; Merceron et al., 1988). In recent years, tosudite has also been reported from Korea (Hwang, 1989; Cho, 1990; Kim, 1991). They report that occurrence of tosudite is almost restricted to the hydrothermal alteration zone of intermediate to acidic igneous rocks. Synthesis of tosudite has been carried out by Matsuda and Henmi (1973) and Ichikawa and Shimoda (1976).

In 1982, the Clay Minerals Society Nomenclature Committee (Bailey et al., 1982) recommended that the name tosudite is valid for a 1:1 regular interstratification of chlorite and smectite that is dioctahedral on average, having a total octahedral population between 6.0 and 7.0 on the basis of $O_{20}(OH)_{10}$ for the assemblage and a d(060) value between about 1.49Å and 1.506Å. This allows combination of di,dioctaheral chlorite (donbassite) with either dioctahedral of trioctahedral smectite and of dioctahedral smectite with either di,trioctahedral chlorite (sudoite of cookeite) or tri, dioctahedral chlorite (unknown to date).

The Sungsan mine where tosudite occurs as a hydrothermal alteration mineral of volcanoclastic rocks, is located 27 km south of Mokpo City and 25 km west of Haenam, Jeonranam-do, Korea. The mine produces monthly about 5,000 tons of agalmatolite and porcelain clay. Agalmatolite is mainly composed of dickite, whereas porcelain clay consists of microcrystalline quartz and dickite. Tosudite is chiefly associated with agalmatolite.

The present paper describes the occurrence of tosudite consisting of di,dioctahedral chlorite and dioctahedral smectite (beidellite), and its mineralogical characteristics in comparison with earlier ones.

EXPERIMENTAL DETAILS

Nearly pure samples of tosudite were prepared by hand-picking, sedimentation and centrifugation. X-ray powder diffraction (XRD) analysis was done using a Rigaku model RAD 3-C and Ni-filtered C_uK_α radiation. Oriented specimens were examined after four separate treatments; (1) air-drying at room temperature, (2) ethylene glycol (EG) saturation, (3) glycerol (GL) saturation, and (4) heating at 110℃, 300℃, 500℃ and 550℃ for each 3 hour. XRD patterns of tosudite were compared with those calculated by the NEWMOD program (Reynolds, 1985).

Electron microprobe analyses were carried out on a few samples using a JEOL JXA-733 superprobe equipped with a LINK AN 10000 energy dispersive X-ray (EDX) analyzer. Lithium was separately analysed by a VG Elemental model PQ II Plus inductively coupled plasma-mass spectrometer (ICP-MS).

Differential thermal analysis (DTA) and thermogravimetric (TG) analysis were performed simultaneously using Rigaku model TSS Thermoflex. Infrared (IR) absorption spectra were recorded with a Perkin-Elmer model 283B spectrophotometer. KBr pellet method was used over the range of 4,000 to 200 cm^{-1} under the conditions of scan time 6 minutes, slit program 6 and expansion 1.

OCCURRENCES

The Sungsan mine is one of the famous agalmatolite deposits in southern Korea. The agalmatolite deposits of this mine are the hydrothermal alteration product of volcanoclastic rocks of Late Cretaceous Period. They consist chiefly of clay minerals such as dickite, illite and tosudite. Tosudite is predominantly found in the alteration zone of rhyolitic tuff of the Hwangsan Formation (Cho, 1990). It is also associated with nacrite and/or quartz in cavities within the massive black claystone which consist mainly of dickite and illite (Fig. 1A).

Tosudite shows thin flakes under the polarizing (Fig. 1C) and scanning and transmission electron microscopes (Fig. 1D).

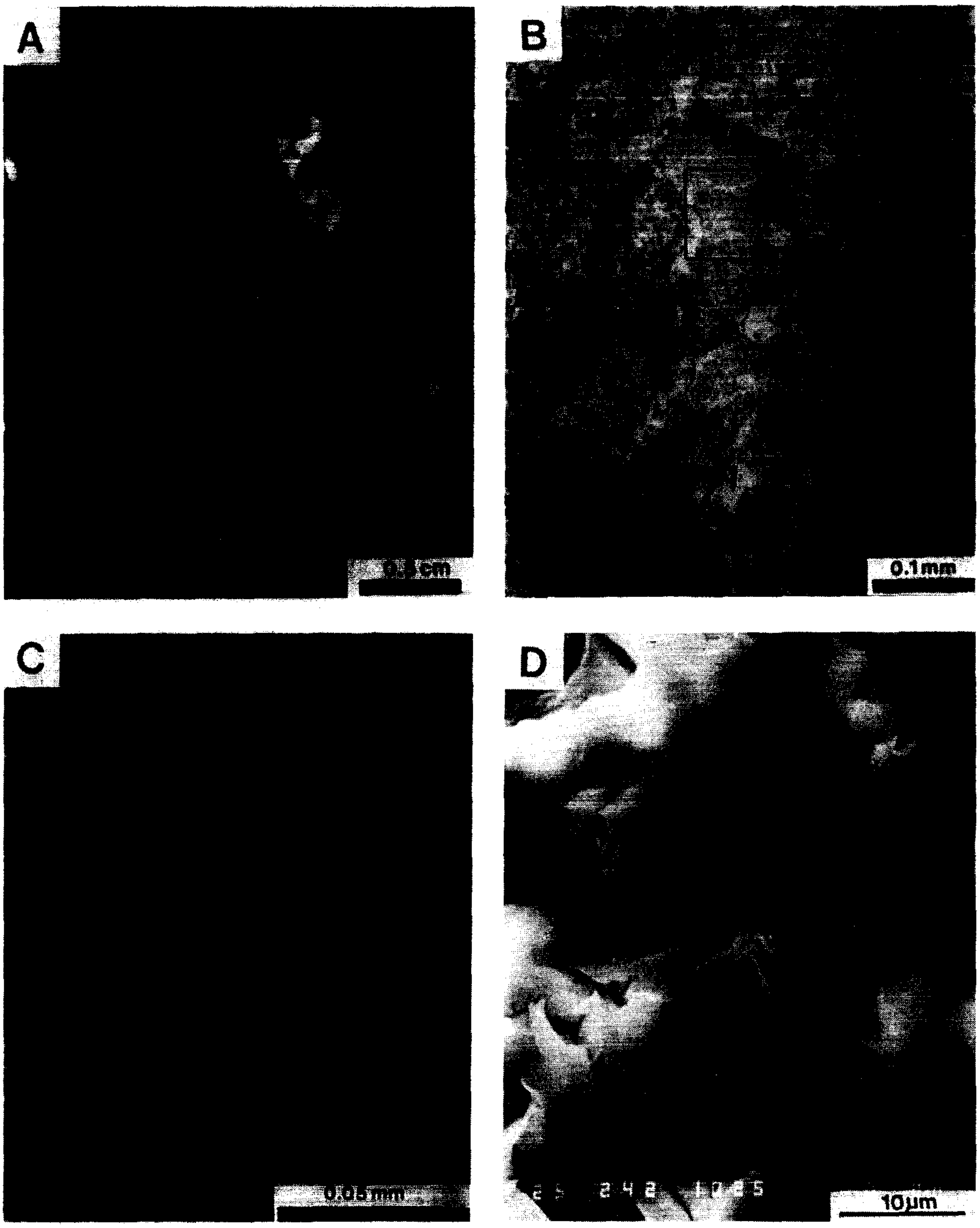

Fig. 1. (A) Tosudite (white) fills the cavities in black claystone (rock slab). (B) Photomicrograph of cavity-filling tosudite (Ts) with quartz (q). (PPL) (C) Enlargement of a square in (B) indicated by arrow. Tosudite comprises flakes. (D) Scanning electron micrograph of tosudite. It shows curled flaky habit.

MIMERALOGICAL DADA

X-ray Powder Diffraction Analysis

XRD pattern of the Sungsan tosudite is shown in Fig. 2, and XRD data are given in Table 1. The characteristic (001) reflection appears at 29.3 Å. The (060) reflection at 1.492 Å indicates that it is dioctahedral. The systematic appearance of high order reflections suggests that it is a regularly interstratified mineral.

After heating at 300℃ for three hours, the (001) reflection changes to a shoulder and the (002) reflection shifts to 13.2 Å. When heated to 550℃, the (001) and (002) reflections shifts to 23.3 and 11.9 Å, respectively. Ethylene glycol treatment causes (001) reflection to shift from 29.3 Å to 31.3 Å. After treatment with glycerol, the 29.3 Å reflection shifts to 32.0 Å.

The experimental XRD patterns of tosudite correspond to the theoretically calculated XRD patterns of tosudite (a regular interstratification of donbassite and dioctahedral smectite) using NEWMOD program (Reynolds, 1985), as shown in Fig. 2B.

Chemical Analysis

Chemical analysis of tosudite from the Sungsan mine is given in Table 2 together with data from other localities. The analysis is average for 8 points analyses. The structural formula of tosudite calculated on the basis of 50 oxygens (= $O_{40}(OH)_{20}$) is:

Interlayer cations: $K_{0.73}\ Na_{0.02}\ Ca_{0.07} + nH_2O$

'Gibbsite sheet': $Li_{0.52}\ Mg_{0.08}\ Mn_{0.01}\ Fe^{3+}_{0.07}\ Al_{4.33}\ (OH)_{12}$

Silicate layer: $Al_{8.00}\ (Si_{13.23}\ Al_{2.77})\ O_{40}(OH)_8$

Comparing the chemistry of tosudite from the Sungsan mine with other reported data, the Sungsan tosudite contains more Al and K, and less Fe, Mg, Ca and Na than the others. It also contains considerable amounts of lithium. Lithium is presumed to be present in the gibbsite sheet as shown by Nishiyama et al. (1975) and Ichikawa and Shimoda (1976).

As shown in the structural formula, the octahedral cations per formula unit of tosudite is about 13.01. The two octahedral sites of the chlorite component appear to be dioctahedral (di, dioctahedral). The interlayer cations are composed mainly of K with minor amounts of Ca and Na.

Table 1. X-ray powder diffraction data of tosudite from the Sungsan mine after various treatments (unit: Å).

001	A	B	C	D	E	F	G
001	29.3	31.3	32.0	28.9	27.2[s]	23.4	23.3
002	14.2	15.4	15.2	13.8	13.2	11.9	11.9
003	9.5		9.5	9.2	8.4[b]		
004		7.8	8.0			5.8	5.8
005	5.70	5.70	5.70	5.69	4.79	4.68	4.69
006	4.78	5.16	5.29	4.67		4.49	4.48
	4.44		4.74	4.46	4.47		
007		4.47	4.55				
008				3.49	3.45	2.93	2.91
009		3.46	3.51		2.88		
00,10	2.85			2.76			
00,11		2.83	2.87				
060	1.492						

A, air-dry; B, ethylene glycol saturation; C, glycerol saturation; D, heated at 110℃ for 3 hrs; E, heated at 300℃ for 3 hrs; F, heated at 500℃ for 3 hrs; G, heated at 550℃ for 3 hrs. s, shoulder; b, broad.

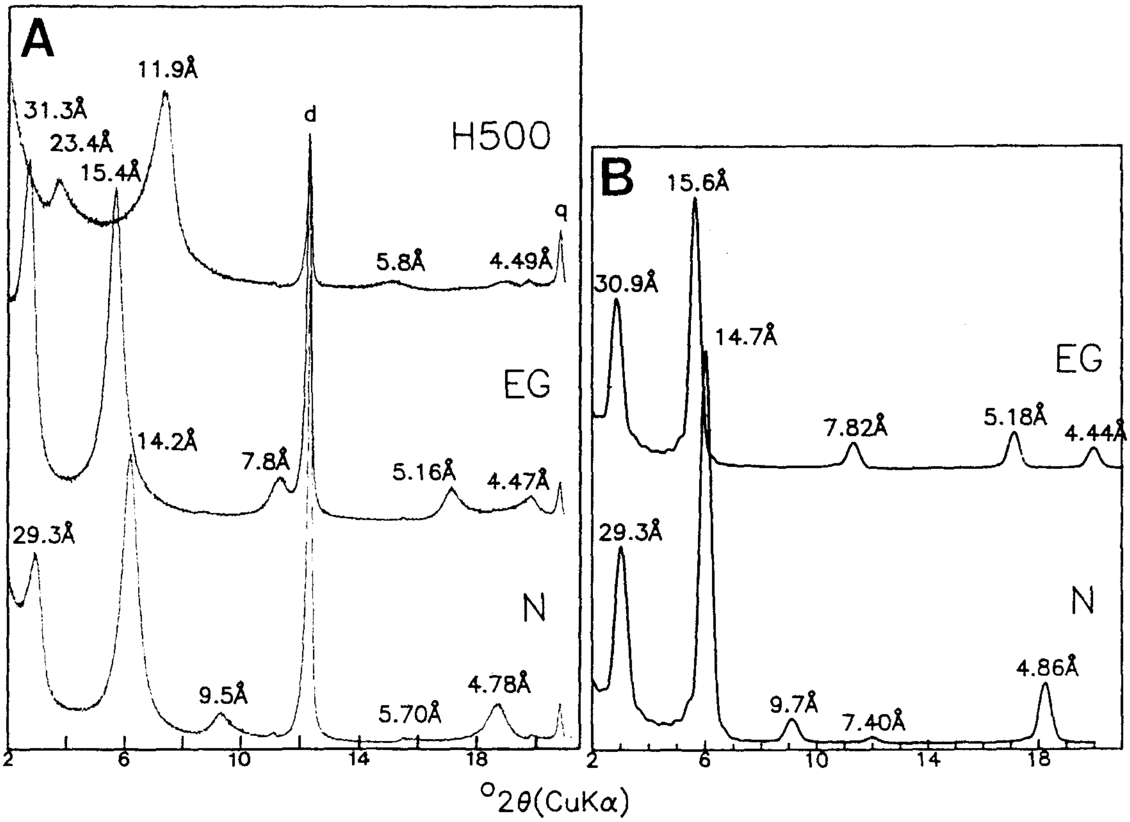

Fig.2. X−ray powder diffraction patterns of tosudite. (A) XRD patterns of tosudite from the Sungsan mine. N, air dry; EG, ethylene glycol saturation; H500, heated at 500℃. d, dickite; g, guartz. (B) Calculated XRD patterns of tosudite (regular interstratification of donbassite and dioctahedral smectite) using the NEWMOD program (Reynolds, 1985).

The Sungsan tosudites are plotted around the hypothetical tosudite composition between ideal donbassite and beidellite (Fig. 3), which is consistant with the XRD data. The chemical analysis suggests that the Sungsan tosudite is a Li-bearing regularly interstratified mineral composed of di, dioctahedral chlorite (donbassite) and K-rich beidellite layers.

Thermal Analysis

DTA and TG curves of the Sungsan tosudite are shown in Fig. 3. The endothermic peak at 45℃ is due to the dehydration of the interlayer water and the other one at 480℃ is due to the dehydroxylation of the gibbsite and silicate layer (Ichikawa and Shimoda, 1976). Dioctahedral chlorite shows one broad endothermic peak due to dehydroxylation at about 500℃, whereas tri-octahedral chlorite shows two sharp endothermic peaks from 600℃ to 850℃ (Smykatz-Kloss, 1974). The dehydroxylation temperature of tosudite at 480℃ suggests that chlorite component in tosudite is dioctahedral.

TG curve shows two distinct steps of weight loss. The first 6.0 % loss of weight below 300℃ is due to the dehydration and the second 8.5 % loss of weight between 350℃ and 750℃ due to dehydroxylation.

Table 2. Chemical analyses of tosudites from the Sungsan mine and other localities (wt. %).

	1	2	3	4	5	6	7
SiO_2	43.30	42.14	41.60	44.20	43.43	39.94	39.74
Al_2O_3	41.90	37.38	36.40	36.65	35.35	33.17	35.87
TiO_2					0.07	0.74	0.01
Fe_2O_3	0.29*	0.30	1.82	1.24	0.35	1.34	0.98
FeO						0.18	2.77
MgO	0.18	0.08	0.29	0.54	0.17	6.44	3.08
MnO	0.04				0.27		
CaO	0.22	1.65	0.38	2.56	0.27	1.30	0.06
Na_2O	0.03	0.15	0.14	0.12	0.28	0.52	0.12
K_2O	1.88	1.40	0.38	0.58	1.90	0.24	0.62
Li_2O	1.42**		1.04	0.60	1.60		0.51
Total	88.26	83.10	82.05	86.49	83.69	83.87	83.76
Numbers of cations on the basis of 50 oxygens							
Si	13.23	13.72	13.60	13.83	13.99	13.06	13.03
Al^{IV}	2.77	2.28	2.40	2.17	2.01	2.94	2.97
ΣTet.	16.00	16.00	16.00	16.00	16.00	16.00	16.00
Al^{VI}	12.33	12.05	11.63	11.34	11.41	9.84	10.89
Ti					0.02	0.18	0.00
Fe^{3+}	0.07	0.07	0.45	0.29	0.08		0.24
Fe^{2+}						0.33	0.76
Mg	0.08	0.04	0.14	0.25	0.08	0.05	1.02
Mn	0.01				0.07	3.14	
Li	0.52		1.37	0.75	2.07		0.67
ΣOct.	13.01	12.16	13.59	12.63	13.73	13.54	13.58
Mg							0.48
Ca	0.07	0.58	0.13	0.86	0.09	0.46	0.02
Na	0.02	0.09	0.09	0.07	0.17	0.33	0.08
K	0.73	0.58	0.16	0.23	0.78	0.10	0.26
ΣCharge	0.89	1.83	0.51	2.02	1.13	1.35	1.34

1, Sungsan mine, korea (this study, 1991); 2, Takatama, Japan (Shimoda, 1969); 3, Tooho, Japan (Nishiyama et al., 1975); 4, Hokuno, Japan (Ichikawa and Shimoda, 1976); 5, Montebras, France (Creach et al., 1986); 6, Kamikita, Japan (Sudo and Kodama, 1957); 7, Huy, Belgium (Brown et al., 1974).
* Total Fe as Fe_2O_3, * * Li_2O determined by ICP-MS.

Infrared Absorption Analysis

IR absorption data of the Sungsan tosudite are given in Table 3 together with those from other localities. The absorption band at 3620 and 3520 cm^{-1} are attributed to the OH stretching vibration in the silicate layer and the 'gibbsite layer, respectively (Ichikawa and shimoda, 1976).

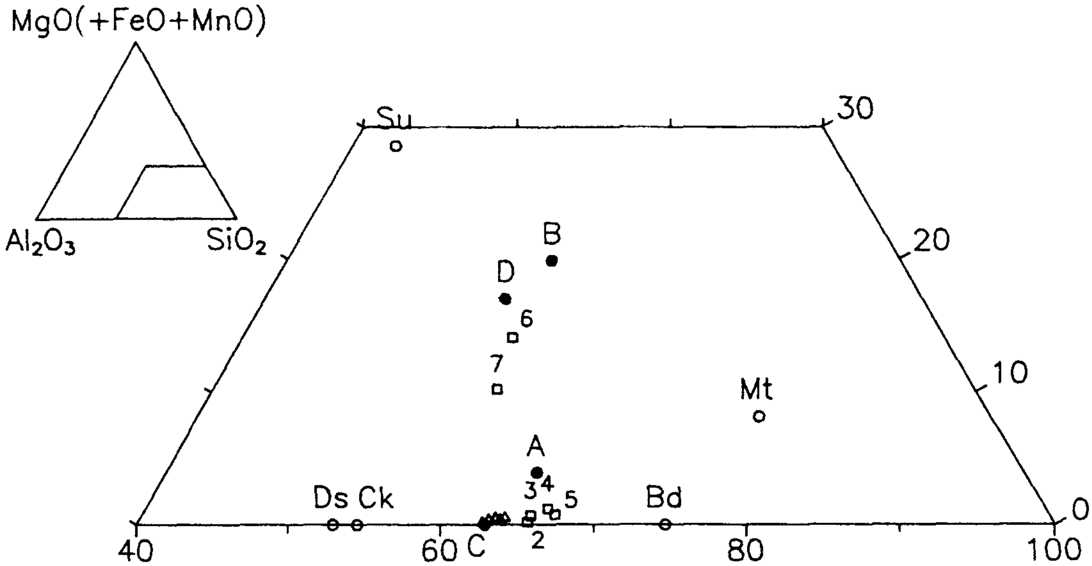

Fig. 3. Plot of mole proportions of SiO_2, Al_2O_3 and MgO (+FeO+MnO) for tosudite and related minerals. Open circles: ideal dioctahedral smectite or dioctahedral chlorite (Bd, beidellite; Mt, montmorillonite; Su, sudoite; Ds, donbassite; Ck, cookeite). Closed circles: hypothetical tosudite compositions between ideal dioctahedral chlorite and smectite (A, montmorillonite and donbassite; B, montmorillonite and sudoite; C, beidellite and donbassite; D, beidellite and sudoite). Triangles: tosudite from the Sungsan mine. Squares: tosudite from other localities (Numbers 2—7 are same as in Table 2).

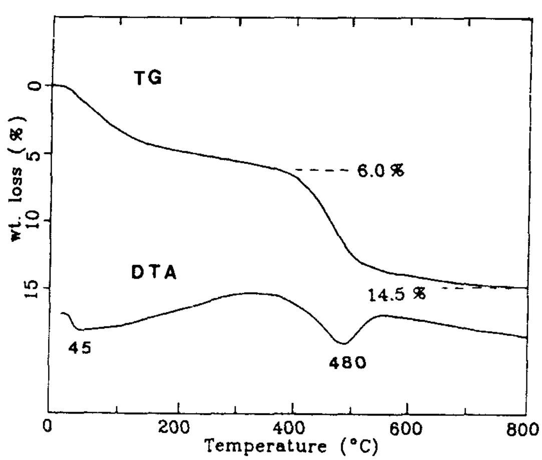

Fig. 4. TG and DTA curves of tosudite from the Sungsan mine.

The band at about 3345cm^{-1} is due to the interlayer water molecules (Shimoda, 1969). The bands at about 1010, 530 and 465cm^{-1} due to the Si-O vibration (Shimoda, 1978). The data are closely similar to those of tosudite composed of di, dioctahedral chlorite and beidellite, but different slightly from those of tosudite with di, trioctahedral chlorite as a component layer.

DISCUSSION AND CONCLUSIONS

The Sungsan tosudite contains a considerable amount of Li_2O (0.42%) and very small amounts of Fe_2O_3 and MgO. Shimoda (1978) and Merceron et al. (1988) pointed out that a continuous solid solution is possible for tosudite minerals between donbassite and cookeite. Merceron et al. (1988) suggested that, if the chlorite layer in tosudite exhibits a donbassite-cookeite solid solution, the Li contents in tosudite be expected to distribute along line B in Fig. 5. Tosudites having smaller Fe+Mg contents are distributed along line B. On the other hand, tosudites containing larger amounts of Fe+Mg are plotted deviation from line B. The latter may be Li-bearing sudoite as a component. Since the Sungsan tosudite contains very small amounts of Fe and Mg, it is plotted near line B. If we consider lithium content in tosudite, 1/4 of the chlorite layer of the Sungsan tosudite is assumed to be Li-bearing cookeite composition. The chemical analyses of tosudites from the Sungsan mine as well as from other localities indicate that the formation of Li-bearing tosudite requires Li in circulating hydrothermal solutions. At the Sungsan mine, the source of Li were uncertain, yet.

Our knowledge of the physicochemical conditions of formation of Li-bearing tosudite is still incomplete. Matsuda and Henmi (1973) observed the transformation of a randomly interstratified illite/smectite to tosudite in pure water at 360℃ and 1 kbar. Ichikawa and Shimoda (1976) also synthesized a tosudite from interstratified illite/montmorillonite at 450℃ and 400 atm. Maksimovic and Brindley (1980) demonstrated that the concentration of Si and Al due to pH decrease is necessary for the crystallization of tosudite. Recently, Foord et al. (1986) specified possible thermal stability field for Li-tosudite, based on the synthetic studies of Eberl (1978a, 1978b). They suggested that Li-tosudite be formed at somewhat lower than 350℃~400℃. All these facts suggest that the formation of tosudite occurs at relatively high temperature. In the Sungsan mine, such conditions probably existed during the intermediate hydrothermal event between the early stage hydrothermal alteration, resulting in the forma-

Table 3. Infrared absorption data of tosudite.

(wavenumber: cm^{-1})

1		2		4		5		6	
		3670	Inf	3670	Inf	3670	Inf	3660	Inf
3620	S	3640	S	3630	S	3630	S	3630	S
3520	W	3545	S	3535	S	3540	S	3540	S
3360	M	3390	SB	3375	M	3400	SB	3380	SB
1640	W	1640	M	1625	M				
1400	W	1404	W						
		1055	Sh						
1035	SB	1035	SB	1030	S	1040	SB	1030	SB
1010	S			1005	S				
940	Sh			940	W	940	Sh		
920*	Sh	950	Inf					940	Inf
830	W			830	W	825	W		
790*	W	820	W					827	W
750	S			750	S	750	W		
720	Sh	750	S						
		735	Sh			708	S		
625	M	705	Sh	629	M	630	Sh	704	S
530	S	630	Sh	535	S	532	M		
510	Sh	544	S			510	Sh	540	S
465	S	523	Sh	465	S	470	S		
405	W	478	S	425	M	420	M	478	S
345	S	415	M			340	S	408	S

Numbers 1−6 are same as in Table 2. *, absorption due to nacrite.
S, strong; M, medium; W, weak; Inf, inflection; Sh, shoulder; B, broad.

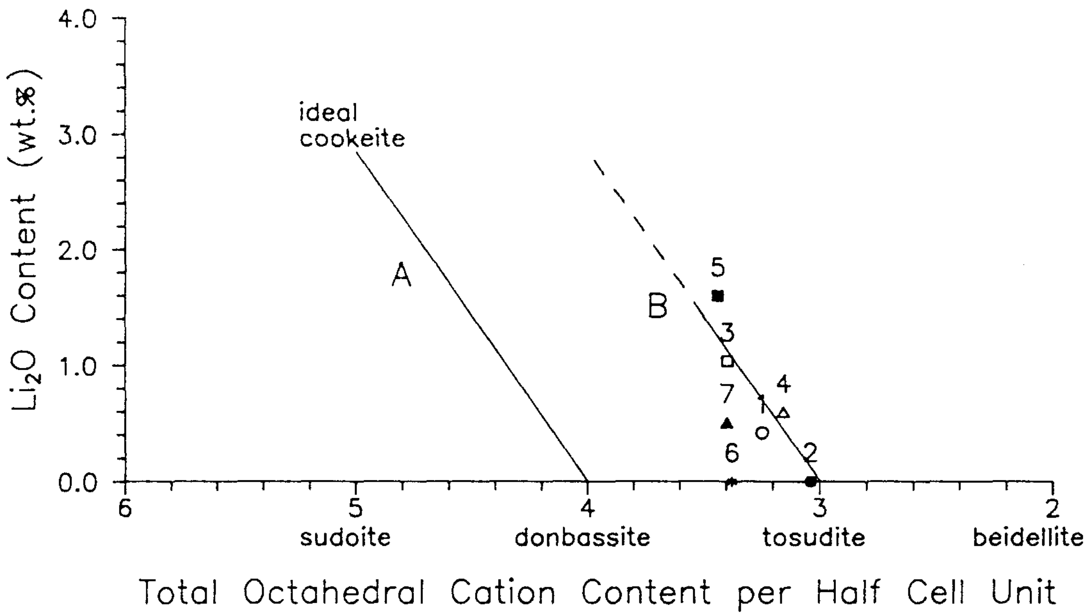

Fig. 5. Diagram showing the relationship between Li_2O content and total octahedral cation content per half cell unit for tosudite (after Merceron et al., 1988). Line A connects the ideal cookeite and ideal donbassite compositions. Line B connects the ideal tosudite composed of donbassite and beidellite and that of cookeite and beidellite. Numbers 2−7 are same as in Table 2.

tion of alunite and dickite (<270℃) and the last alteration of plagioclase to albite (>110℃) (Cho, 1990).

XRD, IR, thermal and chemical analyses of tosudite from the Sungsan mine suggest that it is a 1:1 regularly interstratified mineral of di, dioctahedral chlorite (donbassite) and dioctahedral smectite (beidellite). It contains a considerable content of lithium which is assumed to be in the gibbsite sheet. Potassium is the main interlayer cation in the smectite layers.

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