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Compositional variation of aeschynite-group minerals in the Bayan Obo Nb-REE-Fe ore deposit, Inner Mongolia, China

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Abstract: The aeschynite-group minerals occurring in an aegirine-fluorite-dolomite- aeschynite vein in the Bayan Obo Nb-REE-Fe deposit show complex compositional zoning in BSE images. Microprobe analyses revealed that the concentrations of REE_2O_3 are globally high in aeschynite-group minerals, ranging from 36.72 to 40.32 wt.%. Four species were identified in the same sample, these being aeschynite-(Ce), aeschynite-(Nd), nioboaeschynite-(Ce) and nioboaeschynite-(Nd). The correlations among cations suggest two special coupled substitutions with charge-balanced described by the exchange vectors as $CaNb(Nd,Sm,Pr)_{-1}Ti_{-1}$ and $LaNbTh_{-1}Ti_{-1}$. The chondrite-normalized REE patterns are hump-shaped with the maximum at Pr or Nd. The chondrite-nomalized La/Nd ratios vary in the range ~0.15 to 0.40. It indicates that at Bayan Obo aeschynite minerals are relatively strongly La-depleted and Nd-enriched. The compositional variations in aeschynite-group minerals were controlled by the crystal chemistry and affected by fluid composition.

Key-words: aeschynite-group, microprobe analysis, compositional variation, Bayan Obo.

Introduction

Aeschynite minerals are a group of complex oxides composed of rare earth elements, thorium, calcium, niobium, titanium and tantalum. Generally aeschynite-group minerals are uncommon globally (Rosenblum & Mosier, 1975), but they are widespread and among the main niobium source minerals in the giant Bayan Obo niobium-rare-earths-iron (Nb-REE-Fe) ore deposit, Inner Mongolia, China. Four mineral species and two varieties of the aeschynite-group, *i.e.* aeschynite-(Ce), aeschynite-(Nd), nioboaeschynite-(Ce), nioboaeschynite-(Nd) (Zhang & Tao, 1986), Ti-rich aeschynite-(Nd) (Institute of Geochemistry, 1988) and Th-rich aeschynite-(Ce) (Zhang *et al*., 1995), have been found in the deposit. These minerals have been described in terms of their mineralogical characteristics (Zhang *et al*., 1995; Chao *et al*., 1997) and metamictisation (Gong *et al*., 1992; Tao & Zhang, 1994). But the compositional relationships among the aeschynite-group minerals in the deposit have not been studied.

The aeschynite-group is one of the few series known in which the composition ranges from cerium-dominant to yttrium-dominant. This characteristic is in part controlled by the crystal chemistry of aeschynite. In the structure of aeschynite there are polyhedra of suitable sizes to accept both LREE and HREE. Furthermore, there are coupled substitutions at rare-earth site and niobium sites (Miyawaki & Nakai, 1993; Bonazzi & Menchetti, 1999). This means that the content of niobium correlates with the rare earths. The systematic variation of chemical composition of aeschynite-group minerals in the Bayan Obo deposit is therefore of poten-

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tial significance to the exploration of rare earths and niobium resources.

The aim of this paper is to show the chemical zoning resulting from the evolution of physico-chemical conditions during the crystallization of the aeschynite-group minerals at Bayan Obo. The compositional variation of the aeschynite-group minerals is described. The main substitutions that accompany the chemical zoning also are described and investigated.

Samples and analytical methods

The Bayan Obo Nb-REE-Fe deposit is located approximately 50 miles south of the Mongolia border, at 109°57'E and 41°46'N. The deposit is contained within the sediments of the Proterozoic Bayan Obo Group, which consist predominantly of sandstones and slates, with the exception of the H8 dolomite marble unit, which hosts the vast majority of the ore. The paragenesis of the deposit is extremely complex from deposition, through metamorphism and mineralization, to the intrusion of Hercynian granitoids to the south of the deposit (Chao *et al*., 1997). It is suggested that there exist four metallogenetic periods in the whole mineralizing process of the deposit as follow: (1) Middle Proterozoic (1600~ 1200 Ma); (2) Late Proterozoic (800~ 600 Ma); (3) Caledonian (500~400 Ma); (4) Hercynian (340~270 Ma) (Zhang *et al*., 1994; Ren *et al*., 1994; Bai *et al*., 1996). The two major REE minerals are monazite-(Ce) and bastnaesite-(Ce). The niobium mineralization occurred at the final stage in the Hercynian period (Ren *et al*., 1994; Chao *et al*., 1997; Smith *et al*., 1999). The main niobium minerals are columbite, ilmenorutile, aeschynite and fergusonite.

The samples of the aeschynite-group minerals for examination were taken from a single aegirine-fluorite-dolomite-aeschynite vein, which cuts banded aegirine-magnetite-fluorite ores at the East Orebody.

Mineral examinations were carried out using the back-scattered electron (BSE) mode of a Hitachi S2500 SEM, at an accelerating voltage of 15kV, equipped with a Link System energy dispersive X-ray spectrometer. Analyses of mineral chemistry were made using a Cameca SX50 electron microprobe and the method outlined by Williams (1996) for analyses of the rare-earth elements. Count times of 30 s were used for Y, La and Ce, 50 s for Pr, Nd and Sm, and 20 s for Eu, Gd, Dy, Er, and Yb. Background count times were half of those on the element peak. The analyses were undertaken at an accelerating voltage of 15 kV and 20 nA beam current. A defocused 5 μm diameter beam was used in order to minimize the breakdown of hydrous- and fluorine-bearing phases.

The detection limits obtained were calculated based on 3 σ of the mean background count, and are given along with the data. Interferences between X-ray peaks for the REE were corrected empirically using analyses of standard materials, including a range of synthetic, REE-bearing, calcium-aluminum-silicate glasses, containing from 11 to 22 wt.% of an individual REE in each case, BaF_2 and pure manganese metal. Details of the main peak interferences for the REE are given by Williams (1996). Fluorine was analyzed using a natural apatite standard and a count time of 20 s on the $K\alpha$ peak. Uranium and thorium were analyzed using count times of 20 s and 30 s respectively on the $M\beta$ peak, using uranium oxide and a natural Th-rich monazite as standards.

Results

Chemical composition and nomenclature

Aeschynite occurs as anhedral crystals of variable size (up to 5 cm). The BSE image of aeschynite-

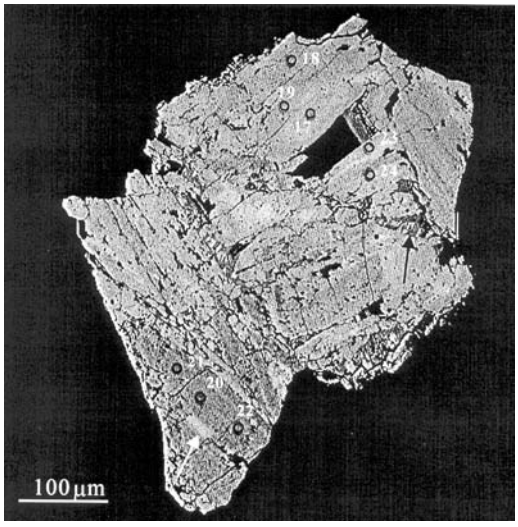

Fig. 1. Backscattered electron images of aeschynite from the East Orebody. The positions marked correspond to analyses. The low-Z area growing along cracks is indicated by the filled arrow. The transitional boundary is indicated by the open arrow.

Table 1. Representative microprobe analyses of aeschynite-group minerals from the Bayan Obo deposit. Values given in wt. %.

	D.L.	25	27	12	33	19	31	24	32
TiO_2	0.17	22.32	23.70	21.69	23.55	19.61	20.81	21.68	21.18
Nb_2O_5	0.42	36.73	33.51	35.99	34.41	41.32	38.56	36.53	36.39
K_2O	0.04	-	-	-	-	-	-	0.05	-
CaO	1.16	1.88	1.14	1.37	1.23	1.90	1.88	1.64	1.80
BaO	0.14	0.31	0.46	0.28	0.31	0.25	0.41	0.28	0.21
SrO	0.26	-	-	-	-	-	0.26	-	0.29
Al_2O_3	0.03	-	-	-	-	-	0.04	-	-
SiO_2	0.08	0.12	0.14	0.13	0.13	0.07	0.11	0.08	0.13
Fe_2O_3	0.16	0.28	0.11	0.24	0.35	0.24	0.36	0.22	0.35
Y_2O_3	0.25	1.20	1.27	1.30	1.98	1.48	1.97	1.27	2.01
La_2O_3	0.26	2.46	1.81	1.43	1.63	2.08	1.86	1.73	1.84
Ce_2O_3	0.18	14.71	14.80	12.89	12.17	13.79	13.25	12.98	12.63
Pr_2O_3	0.21	2.78	3.06	2.77	2.48	2.50	2.48	2.48	2.55
Nd_2O_3	0.24	12.86	14.43	14.04	13.69	12.76	13.28	13.98	13.30
Sm_2O_3	0.23	2.31	2.40	2.67	3.16	2.37	2.56	2.69	2.58
Gd_2O_3	0.11	1.12	1.43	1.88	2.44	1.19	1.51	1.57	1.70
Dy_2O_3	0.27	0.29	0.25	0.58	0.93	0.66	0.72	0.54	0.66
ThO_2	0.26	0.51	0.85	1.92	0.41	0.33	0.47	0.84	0.32
F	0.04	0.06	0.18	0.09	0.20	0.17	0.10	0.15	0.05
Total		99.92	99.55	99.26	99.07	100.72	100.62	98.72	97.97
Structure formulae based on 6(O+F)									
Ti		1.018	1.094	1.007	1.082	0.890	0.947	1.005	0.987
Nb		1.007	0.930	1.004	0.951	1.127	1.055	1.018	1.020
Σ(B+C)		2.024	2.024	2.011	2.033	2.016	2.001	2.022	2.007
K		0.000	0.000	0.000	0.000	0.000	0.000	0.004	0.000
Ca		0.122	0.075	0.091	0.080	0.123	0.122	0.108	0.120
Ba		0.007	0.011	0.007	0.007	0.006	0.010	0.007	0.005
Sr		0.000	0.000	0.000	0.000	0.000	0.009	0.000	0.010
Al		0.000	0.000	0.000	0.000	0.000	0.003	0.000	0.000
Si		0.007	0.008	0.008	0.008	0.004	0.007	0.005	0.008
Fe		0.013	0.005	0.011	0.016	0.011	0.017	0.010	0.016
Y		0.039	0.041	0.043	0.064	0.047	0.064	0.042	0.066
La		0.055	0.041	0.032	0.037	0.046	0.041	0.039	0.042
Ce		0.327	0.333	0.291	0.272	0.304	0.294	0.293	0.287
Pr		0.061	0.068	0.062	0.055	0.055	0.055	0.056	0.058
Nd		0.278	0.316	0.310	0.299	0.275	0.287	0.308	0.294
Sm		0.048	0.051	0.057	0.067	0.049	0.053	0.057	0.055
Gd		0.023	0.029	0.038	0.049	0.024	0.030	0.032	0.035
Dy		0.006	0.005	0.011	0.018	0.013	0.014	0.011	0.013
Th		0.007	0.012	0.027	0.006	0.005	0.006	0.012	0.004
Σ(A)		0.993	0.996	0.989	0.979	0.962	1.011	0.983	1.014
F		0.005	0.018	0.009	0.019	0.017	0.009	0.015	0.005

Note: D.L. = detection limit. "-" = below the detection limit.

group minerals from the Bayan Obo deposit is shown in Figure 1. Variations in brightness of BSE image correspond to differences in the mean atomic number. The compositional zoning is complex and is characterized by different brightness. Some low-Z (Z = mean atomic number) areas visible grow along cracks through the aeschynite crystal. Some boundaries are transitional between high-Z areas and low-Z areas. It indicates that the low-Z areas probably are replacement zones rather than merely representing crack filling.

The chemical composition of aeschynite was examined by electron probe microanalysis. In total 33 spot analyses were performed on 6 individual crystals. Representative analyses are listed in Table 1. They demonstrate that the grains are zoned with respect to Nb, Ti and REE. These elements are the main components in aeschynite. In Figure 2 the

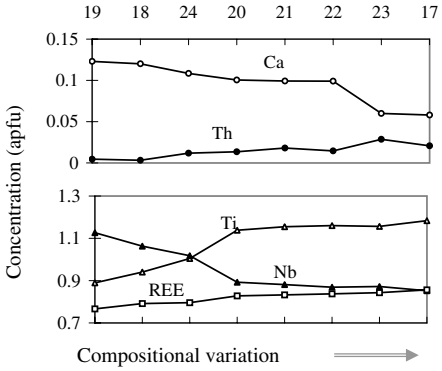

Fig. 2. Compositional variation of aeschynite during progressive vein formation. The diagram was deduced from combination of electron microprobe data and analyses of BSE images. Concentration is shown in atoms per formula unit (apfu). Analysis positions are shown in Fig. 1.

compositional variation of aeschynite during progressive vein formation was deduced from combination of electron microprobe data and analyses of BSE images. The analyses 20, 21 and 22 are from the low-Z areas, while 18, 19 and 24 are from relatively high-Z areas. The high-Z zones have Nb and Ca contents higher, but Ti, REE and Th contents lower than the low-Z zones. This tendency can be considered to be representative of the compositional variation of aeschynite-group minerals with crystallization in the vein from East Orebody. The analyses 17 and 23 are from the high-Z areas, which may have resulted from the increasing concentrations of REE and Th.

Literature data show that aeschynite-group minerals display a wide range of total REE concentrations from ~16% (in Fleischer, 1966 compilation; Edwards *et al.*, 1992) to ~39% (*e.g.* Gieré & Williams, 1992). The results of this study show that the Bayan Obo aeschynite-group minerals generally have high total REE contents from 36.72 to 40.32 wt.% oxide. In spite of the high Nb_2O_5 contents (ranging from ~33 to 41 wt.% oxide), Ta was very low and below the microprobe detection limit (0.3%).

There are many difficulties in naming aeschynite and related minerals because of their generally metamict state and their complex chemical composition (Ewing, 1976). However, euxenite-group minerals are HREE-dominant, while aeschynite-group minerals are usually LREE-dominant (Fleischer, 1966). According to the REE-nomenclature system of Bayliss & Levinson (1988) and the interpretation of the 50% rule in multiple solid solution

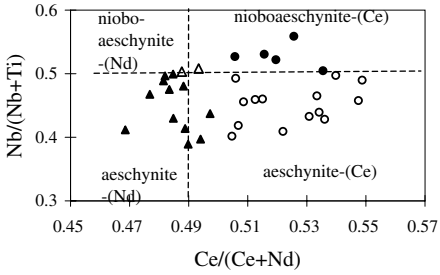

Fig. 3. Nb/(Nb+Ti) *versus* Ce/(Ce+Nd) plot of aeschynite-group minerals from the Bayan Obo deposit, Inner Mongolia, China. Four mineral species can be identified: open circles, aeschynite(Ce); filled triangles, aeschynite-(Nd); filled circles, nioboaeschynite-(Ce); open triangles, nioboaeschynite-(Nd).

series by the CNMMN (Nickel & Grice, 1998), four species in this group in the studied samples can be identified as shown in Figure 3. They are aeschynite-(Ce), aeschynite-(Nd), nioboaeschynite-(Ce) and nioboaeschynite-(Nd).

Crystal chemistry and compositional variation

The formula of aeschynite can be written as $A_{1-x}B_2C_x(O,OH)_6$, where A = REE, Ca, Th, U and Fe; B = Ti, Nb, Ta and W; C = B type cations (Bonazzi & Menchetti, 1999). The A type and C type cations are in 8-fold coordination and B type cations in 6-fold coordination. This structure configuration allows for complex cation substitution to occur with variations in both ionic size and charge. For the aeschynite from Bayan Obo, in B and C sites the Ti and Nb contents correlate very well as shown in Figure 4, thus firmly establishing the substitution between Ti and Nb.

According to the correlations between Ca, Th and REE, using the correlation coefficients of linear regression as references show in Table 2 and Figure 5, in A site the Ca content is negatively correlated to the Th and REE contents. Moreover, the behavior of the

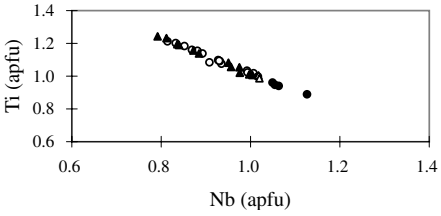

Fig. 4. Ti *versus* Nb plot in atoms per formula unit (apfu). Symbols for aeschynite as in Fig. 3.

Table 2. The correlation coefficients between REE and Ca, Th.

	La	Ce	Pr	Nd	Sm	Gd	Dy	Y	Th	REE
Ca	0.723	0.008	-0.600	-0.873	-0.698	-0.264	-0.198	-0.023	-0.589	-0.753
Th	-0.796	-0.170	0.522	0.712	0.574	0.004	0.035	-0.374	1.000	0.368

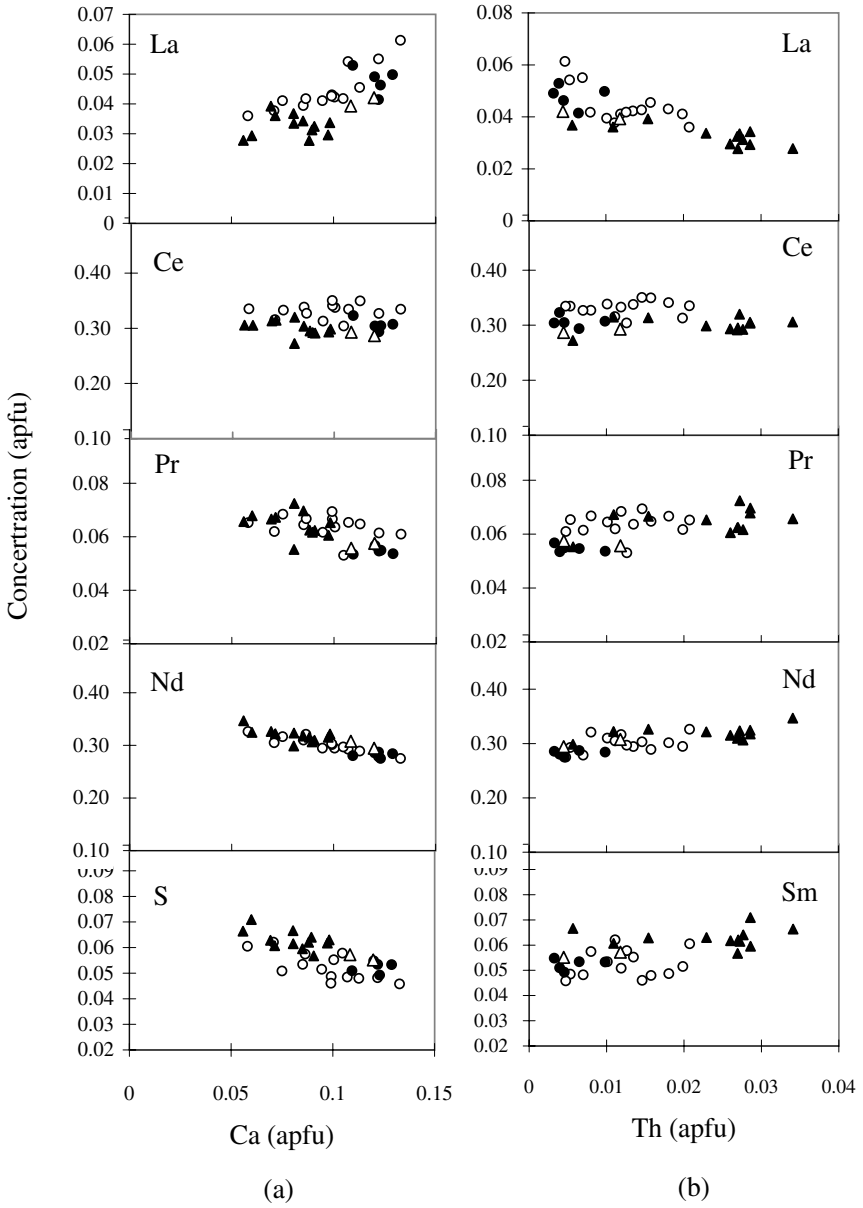

Fig. 5. Correlations (atoms per formula unit) of Ca *vs.* rare earths (a) and Th *vs.* rare earths (b) in aeschynite-group minerals from the Bayan Obo deposit. Symbols for aeschynite as in Fig. 3.

individual rare earths in aeschynite is variable and can be classified as three cases. The Ca content is positively correlated to the La content, but is negatively correlated to the Pr, Nd and Sm contents, and has no distinct correlation with the Ce, Gd, Dy and Y contents. On the other hand, the Th content has a

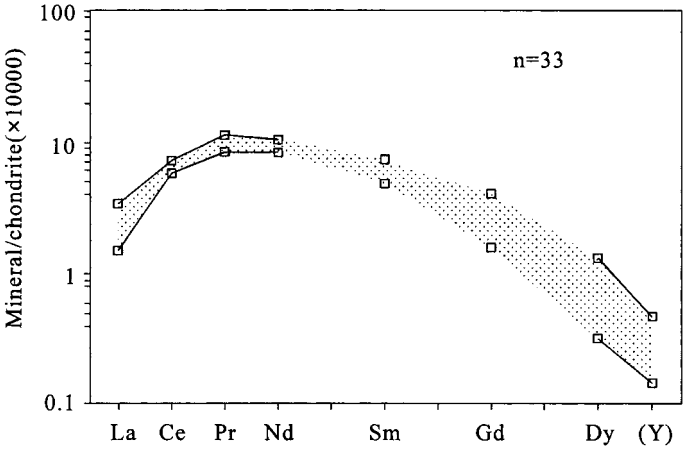

Fig. 6. The range in chondrite-normalized REE distribution for selected light and middle REE in aeschynite from the Bayan Obo. (Chondrite values from Wakita *et al*., 1971).

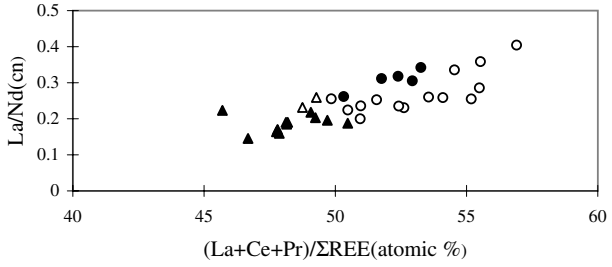

Fig. 7. Plot of the chondrite-normalized La/Nd ratio against the atomic % (La+Ce+Pr)/ (ΣREE) in aeschynite from the Bayan Obo. Symbols for aeschynite as in Fig. 3.

strongly negative correlation with the La content, but positive correlation with Pr, Nd and Sm. On the basis of the compositional variations of aeschynite these correlations suggest the following coupled substitutions with charge-balanced:

$$Ca^{2+} + Nb^{5+} \Leftrightarrow (Nd^{3+}, Pr^{3+}, Sm^{3+}) + Ti^{4+} \quad (1)$$

$$La^{3+} + Nb^{5+} \Leftrightarrow Th^{4+} + Ti^{4+} \quad (2)$$

The chondrite-normalized REE patterns show the variation of rare-earths contents in aeschynite from Bayan Obo (Fig. 6). All of the patterns are hump-shaped with the maximum at Pr or Nd. The atomic ratio of (La+Ce+Pr)/(ΣREE) varies from 0.46 to 0.57 as shown in Figure 7. The chondrite-nomalized La/Nd ratios vary in the range ~0.15 to 0.40. Relative to monazite ((La+Ce+Pr)/(ΣREE)≈0.75–0.89; La/Nd≈1.3–7) and fluorocarbonates ((La+Ce+Pr)/ (ΣREE)≈0.65–0.88; La/Nd≈0.7–5.8) at Bayan Obo (Smith *et al*., 2000), aeschynite are strongly La-depleted and Nd-enriched.

Discussion

Several REE minerals formed during hydrothermal processes exhibit a pronounced zonation (Gieré, 1996). This zonation is interpreted as reflecting fluctuations in the composition of the metasomatic fluid during their formation. The zoning observed for the aeschynite from Bayan Obo indicates that the chemical composition of the fluid must have been variable even during formation of one particular vein zone.

The Bayan Obo aeschynite is very rich in REE_2O_3, particularly in Nd_2O_3 content. This can be better understood by considering whole-rock geochemical data. It is suggested that in Bayan Obo episodes of REE mineralization began from 1600 Ma to 270 Ma (Bai *et al*., 1996). During late-stage hydrothermal activities, Nd mineralization took on increasing importance (Chao *et al*., 1997; Institute of Geochemistry, 1988). The late mineralization consists predominantly of Nd- and more MREE-enriched minerals (Smith *et al*., 2000). The REE minerals enriched in Nd include parisite-(Nd), cebaite-(Nd) and fergusonite-(Nd), besides aeschynite-(Nd) and nioboaeschynite-(Nd) (Zhang *et al*., 1995). These indicate that the high REE_2O_3 and Nd_2O_3 contents in aeschynite at Bayan Obo were related to the hydrothermal fluid composition during formation.

As suggested by Bonazzi & Menchetti (1999),

there are three cation sites in aeschynite: A, B and C sites. The A site is occupied by relatively large cations such as REE^{3+}, Th^{4+} and Ca^{2+}; and the B and C sites are occupied by relatively small cations such as Ti^{4+}, Nb^{5+} and Ta^{5+}. For the isomorphous substitutions between rare-earth ions and non-rare-earth ions, the charge compensation mechanisms found in aeschynite may involve two types (Miyawaki & Nakai, 1993): coupled substitution within the A site, described by the exchange vectors as $CaTh\text{-}REE_{-2}$ and $Ca_2REE_{-1}Na_{-1}$; and coupled substitution between the A and the (B+C) sites, $CaNbREE_{-1}Ti_{-1}$. The complex solid solutions with combinations of both types make it especially difficult to study their chemical compositions and crystal structures.

However, analysis of crystal chemistry for aeschynite from Bayan Obo has established two special types of the coupled isomorphous substitutions in the A and the (B+C) sites: $CaNb(Nd,Sm,Pr)_{-1}Ti_{-1}$ and $LaNbTh_{-1}Ti_{-1}$. These charge compensation mechanisms have important significances in crystal chemistry of rare-earth minerals. It indicates that either Ca or Th substitute selectively rare-earth elements.

In the structure of aeschynite, in the puckered pseudo-hexagonal packing of oxygens the (Nb,Ti,Ta) cations occupy all the octahedral sites. The large (REE, Ca, Th) cations lie in the tunnel of broad holes of the network, forming a distorted eight-coordinated polyhedron (Giuseppetti & Tadini, 1990; Miyawaki & Nakai, 1993). This feature can be explained by the similarity of the ionic radii and coordination numbers of these cations. Among REE the ionic radii of 8-fold coordinated Pr (11.26 nm), Nd (11.09 nm) and Sm (10.79 nm) are closest to that of Ca (11.20 nm) (Shannon, 1976). It is reasonable that the contents of Nd and Ca have the largest negative correlation because of their similar ionic radii. Although the ionic radius of Th (10.50 nm) with 8-fold coordination is close to Gd (10.53 nm) rather than La (11.60 nm), it has been suggested that aeschynite in nature tends to be Ce- and Th-selective (Ewing, 1976; Tao & Zhang, 1994). It is likely that the fluid composition affected the substitution of La by Th in the Bayan Obo deposit.

According to the derivation of vigezzite and thorutite from aeschynite (Burt, 1989), the aeschynite-group minerals at Bayan Obo represent the intermediate members in the solid solution series: vigezzite ($CaNb_2O_6$) – nioboaeschynite [$(REE,Ca)(Nb,Ti)_2O_6$] – aeschynite [$(REE,Ca,Th)(Ti,Nb)_2O_6$] – thorutite ($ThTi_2O_6$). The variations of non-REE cations in aeschynite are from relatively Nb-rich to Ti-rich, as well as from Ca-rich to Th-rich. Thus, it can be inferred that the two important varieties of aeschynite-group, *i.e.* Ti-rich aeschynite-(Nd) (Institute of Geochemistry, 1988) and Th-rich aeschynite-(Ce) (Zhang *et al*., 1995), may represent the products of relatively late-stage mineralization in the Bayan Obo.

Conclusions

Aeschynite-group minerals from Bayan Obo are characterized by complex chemical zonation. They have high concentration of REE_2O_3, particularly Nd_2O_3. The two particular types of substitutions suggest that under some conditions the rare-earth elements should not be viewed as having two subsets, LREE and HREE. It appears that the elemental fractionations can occur between individual rare earths. Neodymium and La have different roles in the substitutions due to the affects of crystal chemistry and fluid composition. Calcium and Th may also substitute selectively for individual rare earth ions. At Bayan Obo the compositional variation of the aeschynite-group minerals may lead to the formation of Ti-rich and Th-rich varieties at the relatively late stage of hydrothermal activities.

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