

Non-metamict orthorhombic AB_2O_6 Y-Nb-Ta-Ti oxides from a pegmatite in Arvoglio, Crana Valley (Toceno, Vigezzo Valley, Northern Italy)

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ABSTRACT. — In the area between Crana and Arvoglio in the Vigezzo Valley (province of Novara, northern Italy), aplite and pegmatite dikes within the «Pioda di Crana South Zone» contain a mineral identified as euxenite on hand-specimen criteria.

One newly-discovered dike is particularly rich in this mineral, together with gadolinite, xenotime, monazite, etc., and five different morphological types of the assumed euxenite were recognized. X-ray diffraction studies show several of the types to be non-metamict, and with electron microprobe analyses, indicate differences in structure and composition. Four of the analysed types are normal euxenites (space group Pcn). One has crystallized with the aeschynite-(Y) structure (space group Pbnm) and has a Ti-rich composition: it thus corresponds to polycrase.

Key words: Y-Nb-Ta-Ti oxides, Aeschynite-(Y), Euxenite, Pegmatite, Vigezzo Valley.

RIASSUNTO. — Nella zona compresa tra Crana ed Arvoglio in Valle Vigezzo (Provincia di Novara) era stato osservato, in alcuni filoni a carattere aplitico o più frequentemente pegmatitico inseriti negli gneiss della «Zona sud della Pioda di Crana», un minerale che sulla base di preliminari risultati analitici veniva identificato come euxenite.

Un nuovo filone qui scoperto si è rivelato particolarmente ricco in questo minerale in associazione a gadolinite, xenotime, monazite, ecc.

In questo particolare filone sono stati riconosciuti almeno cinque tipi morfologicamente differenti di presunta euxenite.

Gli esami di diffrazione ai raggi X mostrano che quasi tutti i tipi contrariamente alla norma, non sono metamictici e, unitamente alle analisi alla microsonda

elettronica, indicano differenze nella struttura e nella composizione.

Quattro dei tipi analizzati sono normali euxeniti (gruppo spaziale Pcn), mentre uno risulta cristallizzato con la struttura dell'aeschynite-Y (gruppo spaziale Pbnm).

Questo particolare tipo ha un alto contenuto in Ti che corrisponde a quello del policrasio.

Parole chiave: Ossidi di Y-Nb-Ta-Ti, Aeschynite-(Y), Euxenite, Pegmatite, Valle Vigezzo.

Introduction

The Vigezzo Valley in the province of Novara, northern Italy, is well-known among mineralogists for the occurrence of pegmatites with various Ta and Nb oxide minerals: columbite (STRÜVER, 1885; COSSA, 1887), strüverite (ZAMBONINI, 1907; PRIOR and ZAMBONINI, 1908), tanteuxenite (ZAMBONINI, 1908; BUTLER and EMBREY, 1959; DE POL and VESCOVI MINUTTI, 1967), tapiolite (ROGGIANI, 1970), microlite, pyrochlore, fersmite and vigezzite (GRAESER et al., 1979). Some of these species were new minerals at the time of their discovery, others were new to Italy.

During the last twenty years, a black mineral with a greasy luster, strongly resembling tanteuxenite in hand specimens, has been collected at several localities in the East Melezzo stream valley (Crana Valley),

which runs parallel to and 4-5 Km W of the Rio Vasca valley (Piano del Lavonchio). The mineral is found in aplitic or, more frequently, in pegmatitic lenses, and shows some differences in morphology from lens to lens. The mineral was identified as euxenite as a result of diffraction and X-ray fluorescence analyses (MATTIOLI, 1977). In 1978, during mineralogical research in the area, the senior author found a new pegmatitic dike containing several interesting minerals, in particular a mineral that resembled the euxenite found previously in the area.

Occurrence

This pegmatite is located in a wooded area between the Melezzo river bed and the Toceno-Arvogno road, approximately 100 metres above the river bed at approximately 1050 m above sea level. It was named the «pegmatite of the wood», to distinguish it from others which have been found in the same valley.

As is the case for all the Crana Valley pegmatites, the dike occurs within the fine-grained, granular, two-mica-bearing alkali-feldspar gneiss of the «Pioda di Crana South Zone» (Penninic Unit) (KNUP, 1958). These rocks lie in contact with the «Orselina Series». They are located north of the Sempione-Centovalli Line, the northern branch of the important Alpine structural feature known as the Insubric Line or Canavese Line, which separates massive rocks (mainly basic and ultrabasic) from schistose gneiss complexes. This setting differs from that of the other pegmatites of the Vigizzo Valley, which are enclosed in rocks of the Orselina Series or in those of the Monte Rosa Zone.

The dimensions of the pegmatite are approximately $3.50 \times 2.50 \times 1.50$ metres; grain size is consistently medium to coarse, with the rarest mineral species concentrated adjacent to the contact with the wall-rock. This part of the pegmatite contains many vugs up to $10 \times 3 \times 2$ cm in size, and at times completely filled with yttrian fluorite. Gadolinite-(Y) (very pure, green and at times transparent) may normally be found in the vugs; the most frequent accessory minerals are

xenotime-(Y), allanite-(Ce), titanite, pyrite and yttrian apatite.

The minerals studied here are found within the main part of the pegmatite, which consists of quartz and potassium feldspar, together with thorium monazite-(Ce) (MANNUCCI et al., 1986), allanite-(Ce), biotite, ilmenite and, more rarely, hematite, beryl and zircon. Grains of magnetite and spessartine are widespread. A few minerals have not yet been identified.

Physical description

The habit indicates orthorhombic symmetry. The characteristics of the different types (Fig. 1) are:

Type 1: black crystals, sometimes reddish-brown, with a very strong luster and granular to conchoidal fracture. Size is very variable, between a few millimetres and two centimetres. The habit is tabular or prismatic; the main forms normally present are {100} and {010} pinacoids terminated by prism faces. Faces of another prism sometimes occur, and rarely a dipyrmaid. The crystals are normally distinct, but in some cases form intergrowths. Type 1 can normally be found in dark quartz in association with magnetite.

Type 2: black crystals with conchoidal fracture and reddish reflections in thin splinters, covered by a thin grey-green layer of dust. Normally in small grains or large crystals (up to $23 \times 12 \times 10$ mm), doubly terminated and

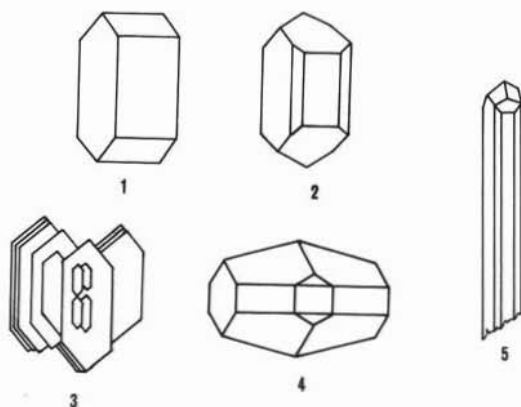


Fig. 1. — Crystal morphologies of Types 1 to 5 for Y-Nb-Ta-Ti oxides from the Arvogno region.

TABLE 1
X-ray powder diffraction data for Y-Nb-Ta-Ti oxides from the Arvorno region

Type 3/4			ASTM 14-643 ¹			Type 1			ASTM 20-1401 ²		
d	I		d	hkl	I/I°	d	I		d	hkl	I/I°
3.0	++		3.01	131	100	3.0	+		2.986	130	75
2.8			2.80	200	40	2.9	++		2.907	112	100
2.58			2.59	002,012	20	2.6			2.767	131	20
2.45			2.44	060,201	30	2.65	+		2.586	200,041	25
1.9			1.907	202	50	2.1			2.11	230	8
1.84			1.839	260	60	2.0			2.029	231	10
1.77			1.782	062	60	1.499			1.498	243,260	14
1.74			1.737	261,330	70						
1.65			1.631	072	10						
1.57			1.566	133	40						
1.50	(+)		1.508	143	10						

d-spacings (Å-units) were measured from Debye-Scherrer films, using a calibrated scale, intensities were estimated visually. Reference patterns with theoretical intensities from the ASTM-index. 1: Synthetic euxenite. 2: Synthetic aeschynite-(Y).

isolated, the main forms are pinacoids {100} and {010} in combination with prism faces and at times a dipyrmaid, and terminated by prism faces. Type 2 is normally found in feldspar in the contact zone of the pegmatite.

Type 3: doubly terminated crystals, up to 2 cm long, similar to type 2, but clustered in a «fan shape» like the classic samples of the Craveggia tanteuxenite.

Type 4: small crystals (only a few millimetres) similar to type 2, but with different dimensional proportions of the faces, and especially with the pinacoid {100} considerably reduced so that the crystals look like «small barrels».

Type 5: crystals very similar to type 2, but very elongated, although commonly very small.

Analytical methods

The analytical work presented here was done to identify the material collected from the pegmatite dike, and to find out whether or not the observed differences in crystal morphology corresponded to variations in chemical composition and/or crystal structure.

X-ray diffraction. Powder diffraction studies were done with 9 cm Debye-Scherrer camera, using Fe-K α radiation and an exposure time of ca. 4 hours. Samples were crushed in an

agate mortar under alcohol, and mounted on a glass capillary with vaseline. The samples were not heat-treated.

Electron microprobe. Fragments of the crystals were mounted in epoxy resin in brass samples holders, polished and coated with carbon before analysis. 25 analytical points were located in 9 grain mounts, using electron backscatter images and qualitative EDS microprobe analyses (to detect compositional zoning and other heterogeneities in the material prior to quantitative analysis). Quantitative analyses were done with a three-spectrometer, fully automated wavelength-dispersive Cameca CAMEBAX electron microprobe with on-line ZAF corrections and data reduction by a PDP 11/34 computer. A maximum of 14 elements was allowed in a single analysis. Standard operating conditions were 15 kV acceleration voltage and 10 nA beam current. The microprobe was standardized with a set of natural and synthetic laboratory standards, including a series of well-characterized 3-element REE oxide glass mixtures (ÅMLI, 1973; ÅMLI and GRIFFIN, 1975).

X-ray diffraction data

Preliminary X-ray diffraction data on

TABLE 2
Mineral compositions for Y-Nb-Ta-Ti oxides from the Arvogeno region

	Type 1		Type 2		Type 3	Type 4			Type 5	Type 3
						core	rim			inclusion
Nb ₂ O ₅	14.58	10.53	24.40	25.46	17.39	19.22	24.38	22.50	18.31	24.93
Ta ₂ O ₅	10.16	21.72	10.02	10.85	26.30	22.28	13.57	7.93	19.40	29.47
TiO ₂	32.22	28.33	22.13	22.35	20.29	22.18	24.68	27.72	24.98	3.79
Y ₂ O ₃	21.13	21.09	14.74	17.65	16.57	21.58	20.74	22.23	21.95	11.66
Yb ₂ O ₃	1.44	0.91	1.84	1.79	1.24	0.93	1.05	2.21	1.57	1.28
Dy ₂ O ₃	2.38	2.14	3.33	3.13	2.64	2.61	3.35	1.83	1.83	2.24
Er ₂ O ₃	1.27	0.85	1.51	1.65	1.09	0.77	1.55	1.43	1.46	0.60
Gd ₂ O ₃	1.42	1.71	1.80	1.56	1.63	1.59	1.94	0.84	1.33	1.33
UO ₂	5.17	2.85	11.73	8.22	5.36	1.96	3.95	6.74	5.48	4.43
ThO ₂	5.59	4.92	3.22	2.91	3.42	1.86	1.67	2.73	2.90	2.84
CaO	0.23	0.45	0.54	0.42	0.92	0.37	0.24	0.15	1.71	0.19
FeO	0.65	0.71	2.28	1.77	1.47	1.40	1.52	0.60	0.80	8.66
MnO	nd	nd	0.31	0.22	0.23	0.23	0.25	0.01	0.07	1.13
Sum	96.24	96.21	97.85	97.98	98.55	96.96	98.88	96.97	100.23	92.52

Structural formulae based on 3.00 cations

Ca	0.015	0.031	0.037	0.028	0.065	0.025	0.015	0.010	0.011	0.015
Fe	0.033	0.038	0.121	0.093	0.081	0.074	0.076	0.031	0.042	0.542
Mn			0.015	0.011	0.013	0.013	0.013	0.001	0.004	0.072
Y	0.673	0.703	0.501	0.589	0.582	0.726	0.665	0.714	0.718	0.465
REE	0.123	0.113	0.171	0.163	0.140	0.119	0.152	0.119	0.120	0.130
U	0.068	0.040	0.167	0.115	0.079	0.028	0.053	0.090	0.075	0.074
Th	0.076	0.070	0.047	0.042	0.052	0.027	0.023	0.038	0.041	0.048
Nb	0.395	0.299	0.704	0.721	0.513	0.549	0.664	0.613	0.510	0.844
Ta	0.165	0.370	0.174	0.185	0.472	0.383	0.222	0.130	0.325	0.600
Ti	1.451	1.336	1.062	1.054	1.007	1.056	1.117	1.255	1.155	0.214
Σch	11.67	11.72	11.86	11.89	11.96	11.86	11.86	11.83	11.89	11.60

The analyses of type 4 are point analyses of a zoned crystal, the others refer to means of several microprobe analyses of homogeneous crystals or parts of crystals. nd= not detected (i.e. below microprobe detection limit). Σch= sum of cation charge, assuming that all Fe is divalent.

unheated material showed that various morphological types give characteristic though somewhat diffuse diffraction patterns (Table 1). Type 1 gives a powder diffraction pattern matching synthetic aeschynite-(Y) (YNbTiO₆, space group Pbnm; ASTM 20-1401), with only small differences in d-values. Types 3 and 4 give patterns corresponding to synthetic euxenite (YNbTiO₆, space group Pcn; ASTM 14-643). Only type 2 is completely metamict. (Type 5 could not be studied, due to the small amount of material available). These preliminary X-ray diffraction results will be followed up with heat-treatment of samples and DTA-studies (ANDERSEN and ALBERTINI, in prep.).

Chemical composition

Representative analyses of the different morphological type are given in Table 2. Analytical totals are generally < 100 wt%; this can be due to the presence of undetected REEs (La, Ce, Nd), each in quantities amounting to tenths of a percent, and/or to the substitution of HO for O. It should, however, be noted that the fluorine content is below the microprobe detection limit (ca. 0.1 wt% F) in all samples studied. All the analyses can be recalculated to structural formulae based on three cations, with good approximation to AB₂O₆ (A = Ca, Fe, Mn, Y, REE, U, Th; B = Nb, Ta, Ti) stoichiometry (Table 2). The recalculated cation

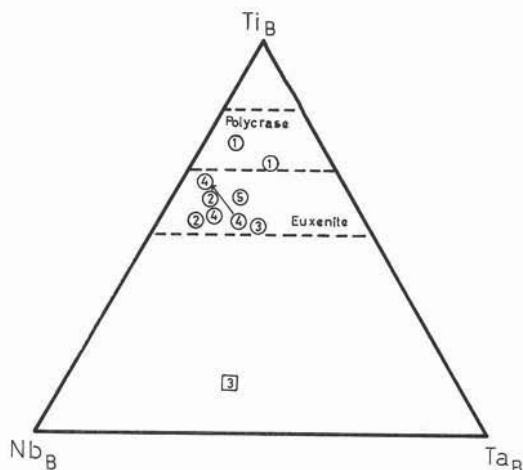


Fig. 2. — Cation distribution at the B-position of the different morphological types, data from Table 2. Square: inclusions in type 3 crystals. Arrow: core-to-rim zoning in type 4.

distributions give small charge deficiencies relative to 6 oxygens ($11.5 < \Sigma \text{ charge} < 12.0$), suggesting the presence of undetected polyvalent cations or of OH^- -substitution for O^{2-} . Oxidation of the small amount of iron present to Fe^{3+} is generally not enough to compensate for the charge deficiency.

As Table 2 shows, the microprobe analyses parallel the X-ray diffraction data in suggesting three different mineralogical varieties among the crystals analysed, morphological types 3, 4 and 5 being compositionally indistinguishable; type 1 is distinctly higher in TiO_2 , whereas type 2 is U-rich. The cation distribution at the A-position is similar in types 1, 3, 4 and 5; however, type 2 has distinctly higher U_A . In terms of substitutions at the B-position, type 1 is characterized by high Ti_B (> 1.3); the other types are indistinguishable, with $Ti_B < 1.2$. Although all types show a limited internal variation in B-cation distribution, only type 4 has a systematic core \rightarrow rim zonation with outwardly increasing Ti_B and Nb_B .

The composition of some small, Fe-bearing inclusions in type 3 crystals is also included in Table 2 and is plotted in Fig. 2.

Discussion

Nomenclature. The systematics of the

orthorhombic AB_2O_6 Y-Nb-Ta-Ti oxides are complex and still poorly understood. This is mainly due to the complex substitutional possibilities and the metamict nature of most naturally occurring specimens (EWING and CHAKOUMAKOS, 1982; CLARK, 1983). Table 3 gives a summary of the different mineral species which have been suggested. The original descriptions (e.g. BRØGGER, 1906) were based on crystal morphology and bulk chemical compositions. From such data, BRØGGER, (1906) inferred the existence of two solid solution series of equivalent compositions: euxenite-polycrase and priorite-blomstrandine, with polycrase and blomstrandine as the Ti-rich members. A survey of published chemical analyses has shown that only two different species can be distinguished compositionally: euxenite ($TiO_2 = 19\text{--}25 \text{ wt\%}$) and polycrase ($TiO_2 = 29\text{--}37 \text{ wt\%}$); published analyses of priorite and blomstrandine fall within the compositional range of euxenite. Both euxenite and polycrase represent solid solution series with extended diadochy between Nb and Ta at the B-position (EWING, 1976; EWING and CHAKOUMAKOS, 1982).

Studies of synthetic crystalline equivalents indicate two different crystal structures in the group: the α - $YTiNbO_6$ or euxenite structure (space-group P_{21} , assumed to represent a high-temperature polymorph) and the β - $YTiNbO_6$ or aeschynite structure (space-

TABLE 3
Nomenclature of the AB_2O_6 group

Traditional name	A-cations	B-cations	Recommended name
α-$YTiNbO_6$-structure:			
Euxenite	Y, REE, U	Ti, Nb, Ta ($Ti=1.0\text{--}1.33$) ²	Euxenite ²
Polycrase	Y, REE, U	Ti, Nb, Ta ($Ti>1.33$) ²	Polycrase ²
β-$YTiNbO_6$-structure:			
Aeschynite	Ce, REE	Nb, Ti, Ta	Aeschynite-(Ce)
Priorite ¹	Y, REE	Nb, Ti, Ta	Aeschynite-(Y) ³
Blomstrandine ¹	Y, REE, U, Th	Ti, Nb, Ta ($Ti>1.33$?)	Aeschynite-(Y) ³

References: 1: BRØGGER (1906), 2: EWING (1976), 3: EMBREY and FULLER (1980).

group Pbnm, isostructural with CeTiNbO_6 and representing a low-temperature polymorph) (SEIFERT and BECK, 1965; EWING and CHAKOUMAKOS, 1982). It is generally agreed that priorite has the aeschynite structure (KOMKOV, 1959; EWING and CHAKOUMAKOS, 1982). Polycrase is thought to be isostructural with euxenite (SEMENOV, 1963; EWING and CHAKOUMAKOS, 1982), although this assumption is based on studies on heated samples only. Heating of metamict AB_2O_6 -minerals need not re-establish the original structure; it is important in this context to note that, on annealing at $T > 1100^\circ\text{C}$, the euxenite structure will be stabilized, regardless of the structure of the crystalline precursor of the metamict mineral (SEIFERT and BECK, 1965; EWING and EHLMANN, 1975). Since the compositional differences between «priorite» and «blomstrandine» are apparently not significant, both names have been dropped in favour of aeschynite-(Y) (EMBREY and FULLER, 1980; cf. LEVINSON, 1966).

Classification of the Arvogno minerals. Of the present material, morphological types 3 and 4 show compositions and structures which allow them to be unambiguously classified as euxenite *sensu strictu*. By inference from the microprobe analyses, this is probably the case for types 2 and 5 as well, type 2 being a U-rich variety. The classification of type 1, which has a polycrase composition and an aeschynite-(Y) type structure, is somewhat more problematical. This mineral may be Pbnm-polymorph of polycrase, or polycrase itself, if the assumption of a Pcan-structure for that mineral species should prove incorrect. From the microprobe analysis, the inclusions in the type 3 crystals can be tentatively identified as samarskite (or a compositionally related mineral).

Implications. The minerals studied in the present work represent one of the few known examples of naturally occurring, non-metamict orthorhombic AB_2O_6 Y-Nb-Ta-Ti oxides. Furthermore, this is an uncommon case where both euxenite and aeschynite structures are present in material from a single occurrence. As such, the minerals from the Arvogno locality may be of considerable value in

providing a clue to the systematics of this poorly understood mineral group. From the present data, the existence in nature of two different structural types is confirmed; furthermore, a correspondence between chemistry (Ti_B) and structure is also indicated. It has been suggested that the REE-distribution in the A-position can control the structure (SEIFERT and BECK, 1965). The effect of the REE on the structures of the present minerals cannot be evaluated until we have more complete REE-analyses than those presently available. The correlation of structure and composition in the Arvogno minerals will be addressed in a follow-up study (ANDERSEN and ALBERTINI, in prep.).

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