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ANNEALING STUDY OF METAMICT, ORTHORHOMBIC, RARE EARTH, AB_2O_6 -TYPE, Nb-Ta-Ti OXIDES

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ABSTRACT

Metamict, orthorhombic, AB_2O_6 -type, Nb-Ta-Ti oxides were annealed over the temperature range 400-1100°C in air. Each charge was air-quenched at 50°C intervals and examined by x-ray powder diffraction. The following phases were identified: euxenite, priorite-aeschnynite, pyrochlore, brannerite, Nb_2TiO_7 , rutile and anatase. During annealing, the priorite-aeschnynite phase crystallized as the low-temperature phase (400°C) followed by the formation of the high-temperature euxenite phase (700-750°C). Both reactions show sharp exothermic peaks on DTA. The transition of priorite-aeschnynite to euxenite occurs over the range 550-750°C for yttrium-rich compositions and 900-1000°C for cerium-rich compositions. Usually the cubic pyrochlore phase is present from 400-1100°C. Unit cell parameters measured at 900°C for euxenite average $a = 5.54$, $b = 14.62$, $c = 5.16\text{Å}$; for priorite-aeschnynite at 600°C average $a = 5.21$, $b = 10.94$, $c = 7.39\text{Å}$, and for pyrochlore average $a = 10.34$ at 600°C and $a = 10.32\text{Å}$ at 900°C.

INTRODUCTION

A common approach to the identification and study of metamict minerals has been to attempt to recrystallize them by various methods of annealing. These methods are essentially heat treatments, some in air from 600-1000°C

(Lima-de-Faria 1964) and others in inert atmospheres using hydrogen (Bannister & Horne 1950), helium (Berman 1955), nitrogen (Lima-de-Faria 1964), vacuum (Peacor & Simmons 1972), or under hydrothermal conditions (Bouška & Johan 1972). Rarely are annealing experiments conducted systematically over a wide range of temperatures or at intervals less than 100°C. In addition, published annealing studies seldom are accompanied by pertinent chemical data, so that the initial compositions are unclear. Results of such studies, therefore, are difficult to compare and interpret.

This paper describes the recrystallization of selected natural compositions of the orthorhombic, rare earth, AB_2O_6 -type, Nb-Ta-Ti oxides at 50° intervals over the temperature range 400-1100°C. Of particular interest is the determination of the temperature of the priorite-aeschnynite → euxenite transition.

PREVIOUS WORK

Previous studies of AB_2O_6 -type Nb-Ta-Ti oxides established the general recrystallization sequence and the phases that are expected to form. Mitchell (1972) summarized the pertinent aspects of these studies for euxenite and priorite; Lima-de-Faria (1964) and Vlasov (1966) compiled briefer summaries of x-ray annealing studies of aeschnynite. The results of these annealing studies follow the same pattern. For metamict spe-

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cimens of euxenite and priorite compositions, an orthorhombic phase, isostructural with both priorite and aeschnyrite (the term "priorite-aeschnyrite phase" refers to this low temperature structure and is not indicative of either a priorite or aeschnyrite composition), forms at low temperatures (200-900°C), and an orthorhombic, euxenite phase forms at higher temperatures (450-1100°C). Both the euxenite and priorite-aeschnyrite are commonly accompanied by a cubic, face-centered, pyrochlore ($A_2B_2O_7$) phase. Other phases reportedly formed are brannerite, betafite, fersmite, rutile, anatase, uraninite, Nb_2TiO_7 and UNb_2O_8 . The compositions of pyrochlore, brannerite and betafite are variable. Less work has been completed on the recrystallization pattern of aeschnyrite compositions. In annealing studies of aeschnyrite-(Ce), the priorite-aeschnyrite phase may form at temperatures as low as 700°C, persisting to temperatures of 1300°C. The high-temperature euxenite phase formed between 700 and 1100°C (Seifert & Beck 1965). A cubic phase ($a = 5.15-5.18\text{\AA}$) was reported between 500 and 800°C (Vlasov 1966).

Reported values of the lattice parameter for the cubic Nb-Ta-Ti oxide phase have varied over a wide range. It was identified as a uranium, rare-earth pyrochlore, $U(Nb,Ta)_2O_8$ (Gasparin 1957), as a "pyrochlore-like" structure with $a = 5.07-5.12\text{\AA}$ (Van Wambeke 1970), as a "beta-fite-like phase" with $a = 10.15-10.21\text{\AA}$ (Mitchell 1972) or simply as a "cubic phase" with $a = 10.09-10.24\text{\AA}$ (Gorzhevskaya & Sidorenko 1963, 1964, 1966) or $a = 5.15-5.18\text{\AA}$ (Vlasov 1966). As a further complication, pyrochlore structures allow a wide range of possible compositions. Vlasov (1966) and Bouska (1970) list at least fifteen mineral species with major cationic substitutions in both the *A* and *B* sites of a pyrochlore-type structure. Aleshin & Roy (1962) synthesized twenty pyrochlore-type structures ($a = 10.19-10.51\text{\AA}$) from different starting compositions. Hogarth (1961) showed that betafite and pyrochlore are isostructural but diadochy greatly affects the intensity of *x*-ray reflections and to a lesser extent the cell parameters.

The literature of the differential thermal analysis of metamict Nb-Ta-Ti oxides has been summarized by Mitchell (1972). Metamict Nb-Ta-Ti oxides with euxenite and priorite compositions show at least one of two exothermic peaks. A low-temperature peak may occur between 470 and 540°C and presumably represents the crystallization of the low-temperature priorite-aeschnyrite phase. A high-temperature exotherm may occur between approximately 700 and 780°C and presumably reflects crystallization

of the high-temperature euxenite phase. A broad endotherm below 300°C is a common dehydration feature of annealed metamict minerals.

EXPERIMENTAL PROCEDURE

Seven specimens were selected to represent major orthorhombic, rare earth, AB_2O_6 -type, Nb-Ta-Ti oxide compositions. Chemical analyses of the experimental specimens are listed in Table 1. Their major compositional characteristics are summarized in Table 2. Note that although there is much confusion concerning the nomenclature of this mineral group, for the purpose of this discussion polycrase and blomstrandine are considered chemically equivalent (i.e. Ti-rich).

The unheated samples were amorphous to *x*-ray diffraction except R12 which, on 16 hour *x*-ray exposure, showed a very broad and weak reflection at approximately 3Å corresponding to the strongest reflection of euxenite, priorite and betafite, and R23 (Čech *et al.* 1972) which showed numerous reflections of the orthorhombic aeschnyrite phase and less intense reflections for rutile. The rutile is intimately associated with the aeschnyrite as interlocking grains and is present as an impurity.

TABLE 1. CHEMICAL ANALYSES OF METAMICT ORTHORHOMBIC AB_2O_6 -TYPE Nb-Ta-Ti OXIDES

	R12 ¹	R13 ²	R24 ³	R25 ⁴	R17 ⁵	R23 ⁶	R26 ⁷
CaO	1.00	0.70	1.70	2.40	1.60	0.99	2.55
MnO	-	-	0.20	0.46	-	1.26	0.20
Fe ₂ O ₃	1.30	1.50	3.33	3.89	1.50	1.24	2.56
PbO	0.90	0.80	0.05	0.36	1.00	0.55	0.40
U ₃ O ₈	5.40	3.80	4.40	12.12	13.20	0.25	0.62
ThO ₂	7.70	5.40	5.80	2.85	3.10	2.26	16.98
Y ₂ O ₃			12.70	11.80		1.63	2.51
Gd ₂ O ₃			1.69	0.89		n.a.	n.a.
Dy ₂ O ₃	26.40	30.90	2.15	1.82	22.30	n.a.	n.a.
Er ₂ O ₃			0.02	2.11		n.a.	n.a.
Yb ₂ O ₃			2.32	3.41		n.a.	n.a.
Ce ₂ O ₃			0.25	0.23		13.21	10.82
Nd ₂ O ₃	1.00	1.50	0.95	0.68	1.30	n.a.	n.a.
Sm ₂ O ₃			1.34	0.56		n.a.	n.a.
Zr ₂ O ₃	27.40	32.40	24.90	22.70	23.60	-	-
Nb ₂ O ₅	16.40	19.00	17.72	23.19	24.20	35.72	25.21
Ta ₂ O ₅	4.70	2.00	4.29	0.49	3.00	-	1.21
TiO ₂	29.50	31.90	31.71	25.45	24.50	25.09	22.65
H ₂ O ⁻	0.20	0.40	-	-	0.20	n.a.	0.20
H ₂ O ⁺	4.20	1.30	3.99	3.63	3.20	n.a.	3.20
TOTAL	98.70	99.20	99.53	99.26	99.10	82.20	89.11
R.I.	2.16	2.19	2.13	2.18	2.21	2.24	2.21
S.G.	4.86	4.94	4.838	4.826	5.11	5.18	5.339
VHN ₅₀	730	692	673	641	690		690
Reflect. (546 nm)	13.11	13.57	14.61	13.68	14.79	14.83	13.57

- Blomstrandine, Kåbuland, Iveland, Norway (analyzed by A.E.C. for the late C.O. Hutton).
- Blomstrandine, Mørefjæra, Arendal, Norway (analyzed by A.E.C. for the late C.O. Hutton).
- Blomstrandine, Kåbuland, Iveland, Norway (analyzed by Hongslo & Langmyhr 1960). Totals are for Hongslo & Langmyhr's complete analysis; U₃O₈ is reported as UO₂ and UO₃.
- Euxenite, Eitland, Farsund, Norway (analyzed by Hongslo & Langmyhr 1960). Totals are for Hongslo & Langmyhr's complete analysis; U₃O₈ is reported as UO₂ and UO₃.
- Euxenite, Iveland, Norway (analyzed by A.E.C. for the late C.O. Hutton).
- Aeschnyrite, Luanga Bridge, Zambia (partial electron microprobe analysis by R.C.E.).
- Aeschnyrite, Ilmen Mts., USSR (partial electron microprobe analysis by R.C.E.).

TABLE 2. MAJOR COMPOSITIONAL CHARACTERISTICS OF ORTHORHOMBIC RARE-EARTH AB_2O_6 -TYPE Nb-Ta-Ti OXIDES

polycrase: R12, R13, R24	$\begin{cases} TiO_2 \gg (Nb_2O_5 + Ta_2O_5) \\ ThO_2 > U_3O_8 \\ Y_2O_3 > Ce_2O_3 \end{cases}$
euxenite: R17, R25	$\begin{cases} TiO_2 \approx (Nb_2O_5 + Ta_2O_5) \\ ThO_2 < U_3O_8 \\ Y_2O_3 > Ce_2O_3 \end{cases}$
aeschnyite: R23, R26	$\begin{cases} TiO_2 < (Nb_2O_5 + Ta_2O_5) \\ ThO_2 > U_3O_8 \\ Y_2O_3 < Ce_2O_3 \end{cases}$

Samples were ground under ethyl alcohol until they passed a 300-mesh screen (previous annealing experiments by the author on a metamict yttrialite and rowlandite showed that variations in grain size may produce variable results). In an annealing study of metamict euxenite, Hutton (1961) found no differences in the x -ray pattern if the sample was heated in air or in vacuum; similarly, no difference was observed if the sample was heated as particles or as finely crushed powder. Samples were heated over the temperature range of 400-1100°C at 50° intervals in air for 24 hours. More extended heating times for metamict minerals apparently produce negligible change in the x -ray pattern (Hutton 1961; Gibson & Ehlmann 1970; Mitchell 1973a).

At each 50°C interval, samples were air-quenched and examined by Debye-Scherrer x -ray powder cameras. Films were corrected for shrinkage and an internal standard (NaCl) was used. The x -ray powder data for euxenite and priorite-aeschnyite were indexed and the unit cell parameters were calculated from partial indexing of more critical reflections based on the structural cells determined by Seifert & Beck (1965) and Komkov (1959, 1963a). The cell parameter of the cubic phase was calculated by least squares refinement of Debye-Scherrer data using the Nelson & Riley (1945) extrapolation function.

Differential thermal analysis was made at a constant heating rate of 25°C/min. from 0-1000°C, calibrated against a quartz standard.

RESULTS AND DISCUSSION

The annealing studies of the euxenite, polycrase (= blomstrandine), and aeschnyite compositions are summarized in Figure 1. Because of interferences, particularly between diffraction lines of the cubic pyrochlore and the priorite-aeschnyite phases, and in some cases, because of weak intensities, certain phases were identified

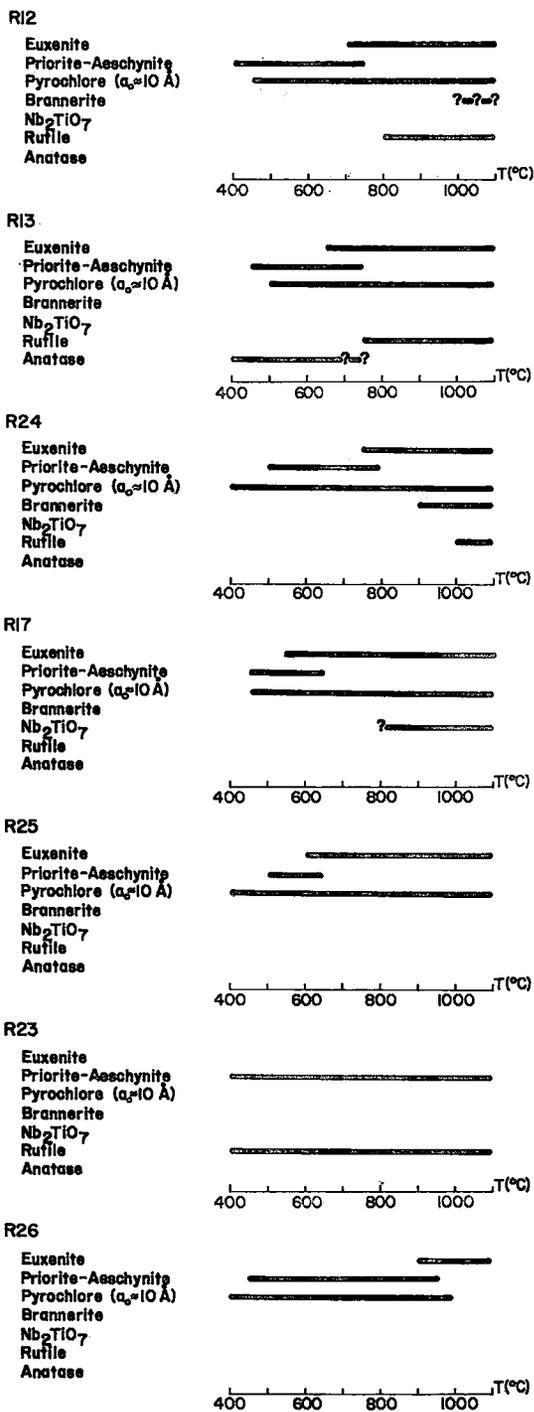


FIG. 1. Summary of annealing data for all samples. Black bars indicate the temperature range over which the phases listed to the left were stable during annealing experiments on the indicated specimens.

tentatively and so indicated by "?" at the appropriate temperature.

The phases observed and their sequence of crystallization for all compositions were similar. The runs differed only in the phase stability ranges and in slight variations in unit cell parameters of the euxenite, priorite-aeschnynite and pyrochlore phases. The polycrase compositions (R12, R13, R24) began recrystallization as priorite-aeschnynite between 400 and 450°C. The priorite-aeschnynite became unstable for all three compositions between 750 and 800°C. Euxenite formed at 650°C and was stable until 1100°C. For euxenite compositions (R17, R25), the priorite-aeschnynite stability range decreased (450-650°C) and euxenite began to form at temperatures as low as 550°C, remaining stable to 1100°C. With aeschnynite compositions (R23, R26), the aeschnynite-priorite phase was stable over a wider range (400-1100°C), and in R23, euxenite was not present in the highest temperature run (1100°C). In six samples, the cubic pyrochlore phase was stable over a wide range (R25, 400-1100°C; R26, 400-1000°C); the pyrochlore phase, however, did not form in R23. Additional phases identified were brannerite, Nb₂TiO₇, rutile and anatase. In at least one sample, R23, the rutile was present as an impurity.

Unit cell parameters for euxenite and aeschnynite-priorite showed only slight variation as annealing temperature increased. In the temperature range of 400-600°C, there were slight decreases in unit cell volume; from 600-1100°C, unit cell parameters were variable. This slight decrease in volume between 400 and 600°C probably reflects the expulsion of H₂O and perhaps oxidation of ferrous to ferric iron; variations above 600°C probably reflect changes in composition. The greatest variation in unit cell parameters was between samples of different compositions at the same temperature. Table 3 contains unit cell parameters for orthorhombic euxenite and orthorhombic priorite-aeschnynite

at 600 and 900°C. The low-temperature priorite-aeschnynite phase formed from polycrase compositions (Ti-rich), R12, R13, R24, have smaller mean volumes than those of the euxenite compositions (Nb-Ta rich), R17 and R25, and much smaller volumes than their Ce-rich aeschnynite analogues (R23 and R26). Similarly, the mean euxenite structure volumes for the Ti-rich compositions are less than for the Nb-Ta-rich compositions. It is difficult to relate volume variation with unique composition changes because the presence of other phases (rutile and pyrochlore) indicates compositions of the euxenite and priorite-aeschnynite structures are not identical to the compositions listed in Table 1. Also, with complex compositions, there are a number of possible A-site and B-site coupled substitutions in which cation volume may either increase or decrease. The volume changes with composition are varied, depending on the rare earth distribution and the change in the average A-site ionic radius. In synthetic studies of simple alpha and beta RENbTiO₆ compositions, unit cell parameters decrease with the decrease in ionic radius of rare earths of increasing atomic number (Komkov *et al.* 1962, 1966; Alexandrov 1963; Seifert & Beck 1965) and increase slightly with the substitution of Ta for Nb (Alexandrov 1963).

There is a slight increase (~0.8%-1.89%) in the volume of the high-temperature euxenite phase over that of the low-temperature priorite-aeschnynite phase of the same specimen. Again, with the natural and complex compositions, it is not possible to evaluate the nature of this volume change because there is no assurance that the compositions of the low- and high-temperature phases are the same. Indeed, for samples R12 and R13, rutile has begun to form between 600 and 900°C, and the increase in volume of the euxenite phase probably results from an increase in the Nb:Ti ratio. The data from studies of synthetic analogues are contradictory. The change in unit cell volume for the beta-YNbTiO₆ → alpha-YNbTiO₆ is +0.85% (Komkov 1959, 1963b; Komkov *et al.* 1962); but for the beta-RENbTiO₆ → alpha-RENbTiO₆, for Z between 62 and 71, there is a slight decrease, -0.5 to -1.3% (Seifert & Beck 1965).

TABLE 3. UNIT CELL PARAMETERS FOR EUXENITE AND PRIORITE-AESCHNYNITE STRUCTURES AT 600°C AND 900°C

		a(Å)	b(Å)	c(Å)	a:b:c	V(Å) ³	phase
600°C	R12	5.15	10.91	7.37	0.472:1:0.676	414.09	P-A
	R13	5.17	10.90	7.27	0.474:1:0.677	409.69	P-A
	R17	5.14	10.87	7.36	0.473:1:0.677	411.22	P-A
	R23	5.37	11.09	7.55	0.484:1:0.681	449.63	P-A
	R24	5.16	10.91	7.32	0.473:1:0.671	412.08	P-A
	R25	5.18	10.89	7.36	0.476:1:0.676	415.18	P-A
	R26	5.30	11.01	7.49	0.481:1:0.680	437.06	P-A
900°C	R12	5.56	14.69	5.17	0.378:1:0.351	422.27	E
	R13	5.54	14.48	5.15	0.383:1:0.356	413.13	E
	R17	5.55	14.70	5.16	0.378:1:0.351	420.98	E
	R23	5.37	11.09	7.55	0.484:1:0.681	449.63	P-A
	R24	5.53	14.63	5.16	0.378:1:0.353	417.46	E
	R25	5.53	14.61	5.17	0.379:1:0.354	417.70	E
	R26	5.30	11.01	7.49	0.481:1:0.680	437.06	P-A

P-A=priorite-aeschnynite structure; E=euxenite structure
Following the method of Bacon (1948) and using NaCl as an internal standard, the estimated error is less than ±0.003Å.

TABLE 4. UNIT CELL PARAMETERS FOR THE CUBIC PYROCHLORE PHASE AT 600°C AND 900°C

	a(Å) at 600°C	a(Å) at 900°C
R12	10.31	10.33
R13	10.27	10.22
R24	10.28	10.31
R17	10.38	10.37
R25	10.43	10.40
R23	10.32	10.25
R26	10.38	10.34

Estimated standard error is less than ±0.003Å.

Structural data for the cubic pyrochlore phase are summarized in Table 4. In this study, *x*-ray powder patterns were indexed and *a* calculated based on a pyrochlore structure with $a \approx 10.00 \text{ \AA}$ (Hogarth 1961). A pyrochlore-type structure with $a \approx 5.00 \text{ \AA}$ was not observed and its previous identification may result from an error in indexing (Pabst 1954) or from the presence of a disordered pyrochlore phase (Alexandrov & Pyatenko 1959; Pyatenko 1960). With an increase in temperature the "*a*" of some samples (R12, R25) increases; but, for others (R13, R17, R23, R25, R26) there is a decrease. The decrease in *a* may result from expulsion of water, oxidation of ferrous to ferric iron or a change in composition (e.g. increase in Ti or Fe³⁺ in the *B*-site; Hogarth 1961). Conversely, the increase in *a* may reflect a decrease in Ti or Fe³⁺ content, perhaps because of the formation of rutile. Changes in the relative intensities of reflections also indicate variations in composition. For pyrochlore formed from euxenite compositions (R17 and R25), intensities correspond more closely to those reported for betafite (uranium pyrochlore) by Hogarth (1961).

TABLE 5. SUMMARY OF DIFFERENTIAL THERMAL ANALYSIS DATA

		Temperature (°C) of exotherms	
R1	aeschninite	Hitterö, Norway	520 740*
R2	aeschninite	Minsk, Urals	770*
R3	aeschninite	Hitterö, Norway	515 750*
R5	polycrase	Minas Geraes, Brazil	540 770*
R6	polycrase	Minas Geraes, Brazil	510 770*
R7	polycrase	Hitterö, Norway	751*
R8	polycrase	Minas Geraes, Brazil	510 770*
R9	polycrase	San Jose de Bujaube, Brazil	710*
R11	priorite	Ahi-Trombe, Madagascar	710*
R12	blomsstrandine	Kåbuland, Iveland, Norway	465 750*
R13	blomsstrandine	Morefjaer, Arendal, Norway	475* 710
R14	blomsstrandine	Iveland, Norway	740* 750*
R15	blomsstrandine	Hitterö, Norway	490* 770
R16	blomsstrandine	Morefjaer, Norway	725* 730*
R17	euxenite	Iveland, Norway	740*
R18	euxenite	Betsiboka Valley, Madagascar	730*
R19	euxenite	Voandelaka & Mandalode, Madagascar	740*
R20	euxenite	Sjaen, Krajerø, Norway	735*
R21	euxenite	Betsiboka Valley, Madagascar	730*
R22	euxenite	Brazil	510 765*
R27	priorite	Mbabana, Swaziland, S. Africa	500* 760
R28	euxenite	Sabine Tp., Nipissing Dist., Ont.	470* 670

*The major exotherm on the pattern

Table 5 contains differential thermal analysis data for 22 specimens. Although the detailed character of the DTA curves is variable, the general shapes fall into two categories: (1) major exothermic peak between 450 and 550°C and (2) major exothermic peak between 650 and 800°C. Curves typical of both types are shown in Figure 2. A broad endotherm below 300°C is present in nearly all cases representing the dehydration of the metamict minerals. The low-temperature exotherm is produced by the crystallization of the priorite-aeschninite phase; the high-temperature exotherm, the euxenite phase. These temperature ranges are in agreement with

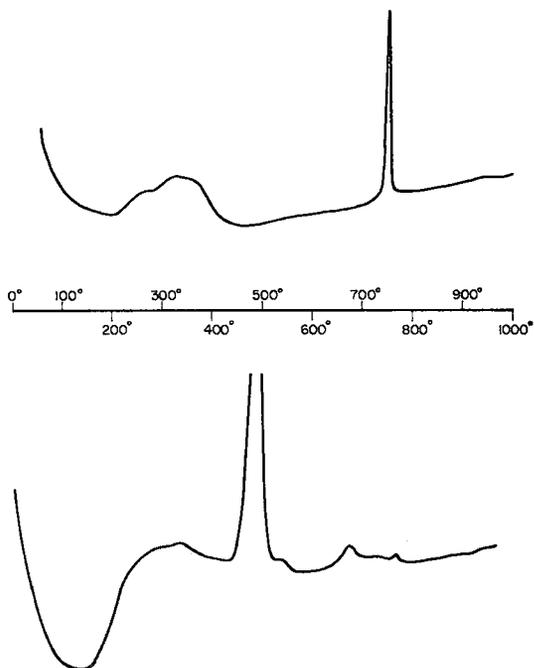


FIG. 2. Differential thermal analysis curves characteristic of two types of metamict orthorhombic, rare-earth, AB₂O₆-type Nb-Ta-Ti oxides. The upper curve shows the high-temperature exotherm associated with the crystallization of the euxenite phase; the lower curve, the low-temperature exotherm associated with the crystallization of the priorite-aeschninite phase.

those noted in the previous *x*-ray annealing study for beginning crystallization temperatures of priorite-aeschninite and euxenite. Hogarth (1961) in annealing studies of pyrochlore and betafite has noted that phases are commonly present below the temperature of the exothermic peak which presumably represents their crystallization, and that above the exothermic temperatures, *x*-ray patterns become more intense although not necessarily sharper. The same phenomenon was noted in this study. The exothermic peaks may be preceded by nucleation and crystallization of the phase in question in small quantity, but at the exothermic temperature the bulk of the phase is formed and *x*-ray diffraction intensities increase.

CONCLUSIONS

During recrystallization, priorite-aeschninite forms as the low-temperature phase ($\sim 400^\circ\text{C}$) and is followed by the formation of the high-temperature euxenite phase ($700\text{--}750^\circ\text{C}$). The transition of priorite-aeschninite to euxenite may

occur over a range of temperatures, 550-750°C, for yttrium-rich compositions and in the range 900-1000°C for cerium-rich compositions. Usually the cubic pyrochlore phase is present throughout the 400-1100°C temperature range.

Despite general similarities, each annealing run is unique in detail. Possible causes of the variations are summarized below:

1. *Degree of metamictness*: If a specimen is completely metamict, recrystallization will not necessarily restore the original crystalline phase; however, if the structure is only partly destroyed during the process of metamictization, the remaining domains of structure may act as nucleation centers and thus determine the phase that forms. In the partly metamict aeschynite, R23, from Zambia, the priorite-aeschynite phase is the first formed and it persists to the highest run temperature (1100°C).

2. *Compositional variations*: For completely metamict specimens, composition will control the stable phase assemblages. The stability field of the aeschynite-priorite phase is increased (conversely the stability field of the euxenite phase is decreased) by either an increase in the ThO_2 : U_3O_8 or Ce_2O_3 : Y_2O_3 ratio. These qualitative generalizations are demonstrated in nature by priorite-aeschynite structures which are characteristically high in ThO_2 and Ce_2O_3 , and by the absence of a cerium-rich euxenite structure. Experimental studies by Alexandrov (1963) on synthetic euxenite and priorite-aeschynite structural analogues show that the structural type which forms is a function of the "average ionic radius" of the A-site cations and of the temperature. As the "average ionic radius" approaches 0.98Å, the compounds become dimorphous and temperature-dependent. Because natural compositions are more complex in rare earth distributions, variations in the temperatures of the priorite-aeschynite → euxenite transition should be expected in annealing studies of natural materials and are, in fact, observed. Further, if alteration occurs after metamictization and changes in composition result, then the non-stoichiometric mixtures may give results substantially different from annealing studies on unaltered material (e.g. formation of anatase, rutile or uraninite). Details of these effects are documented by Van Wambeke (1970).

3. *Experimental variations*: Comparing with previous work, it must be recalled that special environments and variable annealing temperatures have been used. Experimental conditions in this study have been standardized so that variation in phase assemblages are a result of either variation in composition or temperature.

For completely metamict specimens of similar compositions (e.g. euxenite and priorite) annealing data cannot provide unequivocal identification. As pointed out by Mitchell (1973b), even for standardized experimental techniques, recrystallization temperatures of metamict complex Nb-Ta-Ti oxides vary over a wide range depending on (1) variations in chemical composition (and thus the degree of alteration), (2) the degree of metamictization and (3) the structure of the original mineral. Additional data (such as morphology) are required for positive identification of the pre-metamict material.

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