Heat treatment of metamict euxenites, polymignites, yttrotantalites, samarskites, pyrochlores, and allanites.

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Summary. The standard thermal treatments proposed by Lima-de-Faria have been applied to metamict euxenites, polymignites, yttrotantalites, samarskites, pyrochlores, and allanites. In the case of these metamict niobotantalates a face-centred cubic phase is formed at 700° C., and at 1000° C. a more characteristic phase appears combined with the cubic one. In the case of allanites only a slight formation or strengthening of the original structure of allanite appears at 700° C., and at 1000° C. a cubic phase is formed together with other phases.

S TANDARD thermal treatments for metamict minerals were proposed by Lima-de-Faria (1956) to make easier the identification of metamict minerals by X-ray powder photographs. The choice of the conditions for these heat treatments was based on the hypothesis that up to 1000° C. only two crystalline phases, or at the most three, will in general develop on heating. It was also pointed out that in many cases the phase formed at low temperature is cubic, and at 1000° C. a second, more characteristic crystalline phase appears, combined with the first one.

The present paper is intended as a contribution of observational data concerning this problem.

We have applied these standard thermal treatments to a range of metamict niobotantalates: euxenites, polymignites, yttrotantalites, samarskites, and pyrochlores. All these niobotantalates were completely metamict as shown by the powder photographs, which exhibit no lines at all, and for each mineral species at least three samples from different sources were investigated.

On heating the powders in air to 700° C. for 3 hours—the secondary standard thermal treatment—face-centred cubic phases are obtained. For euxenites and polymignites we got a 5·07 Å., for yttrotantalites a 10·28 Å., for samarskites a 5·16 Å., and for pyrochlores a 10·38 Å. (tables I and II). Following Orcel (1956) we think that these cubic phases are probably oxides of one or two of the main elements of the minerals. The cell dimension of ZrO_2 is a 5·07 Å. (Duwez and Odell, 1950), and, since

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TABLE I. X-ray diffraction powder data for polymignite from Frederiksvarn, Norway, and for euxenite from south Norway, heated in air to 700° C. for 3 hours and heated in air to 1000° C. for 1 hour; Fe-$K\alpha$ radiation; I estimated by eye. Also for a euxenite from Ontario, Canada, heated to 1000° C. for 100 hours; Cu-$K\alpha$ radiation (Arnott, 1950). b, broad; vb, very broad. The indices refer to a cubic cell with a about 5·07 Å.

| | Polymignite, Frederiksvarn, Norway. | | | | Euxenite, south Norway. | | | | Euxenite, Ontario. (Arnott, 1950) | |
| | 700°, 3 hr. | | 1000°, 1 hr. | | 700°, 3 hr. | | 1000°, 1 hr. | | 1000°, 100 hr. | |
hkl.	d, Å.	I.	d, Å.	I.	d, Å.	I.	d, Å.	I.	d, Å.	I.
									7·30	1
									5·16	2
							3·62	4	3·66	3
							3·35	1	3·36	2
							3·26	1		
							2·97	10	2·98	10
111	2·92	10	2·94	10	2·92	10	2·93	4	—	
			2·81	3						
							2·77	3	2·77	2
									2·61	¼
							2·58	2	2·58	2
002	2·53	4	2·52	4	2·53	4	2·53	2	2·54	½
							2·42	1	2·43	3
							2·30	1	2·30	1
									2·252	¼
							2·20	1	2·199	1
									2·182	1
							2·11	2	2·106	2
									2·071	¼
									2·025	½
							1·97	1	1·970	½
									1·935	1
							1·89	4	1·889	2
							1·83	5	1·823	4
022	1·79	7	1·82	8	1·79	7b	1·80	6	—	
							1·77	4	1·769	3
			1·75	5						
							1·72	5	1·723	4
									1·679	½
							1·64	5	1·635	3
									1·605	1
									1·583	½
							1·56	3	1·560	1
113	1·53	7	1·55	5	1·53	7b	1·54	6	—	
			1·53	5						
									1·522	¼
									1·501	¼
							1·49	4	1·487	4

TABLE I (*cont.*).

hkl.	Polymignite, Frederiksvarn, Norway. 700°, 3 hr.		1000°, 1 hr.		Euxenite, south Norway. 700°, 3 hr.		1000°, 1 hr.		Euxenite, Ontario. (Arnott, 1950) 1000°, 100 hr.	
	d, Å.	*I*.	*d*, Å.	*I*.	*d*, Å.	*I*.	*d*, Å.	*I*.	*d*, Å.	*I*.
222	1·46	1b	1·48	2b	1·46	1vb	1·46	1	1·457	1
							1·44	3	1·436	2
									1·400	½
									1·391	½
									1·359	1
							1·340	1	1·336	2
									1·311	1
							1·290	1	1·292	1
004	1·267	1b			1·269	1vb	1·280	2	—	
									1·273	½
			1·260	1						
									1·256	¼
									1·241	½
							1·225	1	1·222	2
									1·216	1
									1·203	1
							1·185	1	1·182	2
133	1·167	3b	1·179	5	1·164	2vb	1·174	5	1·173	2
							1·159	2b	1·156	3
024	1·133	3b	1·149	2b	1·135	2vb	1·145	4	1·143	½
							1·118	1	1·119	½
			1·115	1					1·110	1
							1·103	1	1·100	1
									1·091	½
							1·083	2b	1·080	2
							1·057	1vb	1·057	1
			1·050	2						
224	1·034	2vb	1·038	3	1·034	2vb	1·045	3b	1·046	1
							1·034	1vb	1·030	½
							1·020	3vb	1·024	2
									1·015	1
									1·008	½
							1·001	1vb	0·996	1
115 333			0·985	2vb			0·985	3vb	0·986	

polymignite generally has a high Zr content, the cubic phase observed for polymignite might be ZrO_2.

These minerals were heated in powder rather than in fragment form for the sake of better standardization. In fact, if air oxidation does occur it can be complete when powder is used, whilst it may be confined to the outer portion in the case of fragments, consequently leading to differences in results.

On heating the powders in air to 1000° C. for 1 hour—the basic standard thermal treatment—new lines appear in addition to the lines of the cubic phase (tables I and II). These results are similar to those obtained with thorite (Pabst, 1952) who observed that ThO_2 develops first, followed by tetragonal and monoclinic forms of $ThSiO_4$. Attempts to identify euxenite or polymignite by powder photographs from heat treatment at 700° C. alone would lead to ambiguous results (see table I), whereas by heating at 1000° C. the new lines observed are different, and accurate conclusions can be drawn. One of the samples of pyrochlore (from Frederiksvarn, Norway) and a microlite from Virginia, U.S.A., give at 1000° C. only the cubic phase.

X-ray diffraction powder data for samarskites are not included in this paper, because the standard thermal treatments of samarskites lead to results closely similar to those obtained by Lima-de-Faria (1956), heating at 600° C. for 5 hours and 1000° C. for $1\frac{1}{2}$ hours.

In the case of euxenite, Arnott (1950) observed differences on the powder photographs from samples heated to 400° C., 800° C., and 1000° C., and ascribed these differences to an incomplete recrystallization of an orthorhombic phase. On heating euxenite to 1200° C., Berman (1955) also obtained a few strong lines that could not be indexed on the basis of the orthorhombic phase pointed out by Arnott. In fact two phases are formed at 1000° C., one cubic and one orthorhombic, in varying proportions. The powder pattern selected by Arnott for indexing corresponds to heating in air to 1000° C. for 100 hours, which prolonged heat treatment probably made the cubic phase virtually vanish, so that only the orthorhombic phase was obtained. This can be seen in table I: the strong lines of the cubic phase are missing on Arnott's diffraction powder data. Another euxenite, namely a tanteuxenite, studied by Arnott (1950), gives a cubic pattern with a 5·10 Å., which is similar to the author's own results.

We have also applied these heat treatments to several metamict allanites.

X-ray powder patterns were taken of nine unheated allanites from

different localities for a preliminary assessment of the state of the samples. Powders of five of these allanites, ranging from completely to slightly metamict, were heated in air to 700° C. for 3 hours, and only a slight formation of the original structure, or strengthening of this original structure in the case of partially metamict samples, was observed.

The application of the basic standard thermal treatment (1000° C. for 1 hour in air) to the nine allanites yielded very good and similar powder photographs, which seem to be a combination of lines from a cubic phase, a few from the original structure, and a few more from one other phase. The plane spacings printed in italics (table II) fit well to a face-centred cubic phase with a 5·44 Å. Slight differences observed on

TABLE II. X-ray diffraction powder data for yttrotantalite from Ytterby, Sweden, pyrochlore from Langsundfjord, Norway, and allanite from Greenland, heated in air to 700° C. for 3 hours and heated in air to 1000° C. for 1 hour. Cu-$K\alpha$ radiation for yttrotantalite and allanite, Fe-$K\alpha$ radiation for pyrochlore; I estimated by eye. The indices refer to the cubic unit cells mentioned in the text.

Yttrotantalite, Ytterby, Sweden.					Pyrochlore, Langsundfjord, Norway.					Allanite, Greenland.				
700° C., 3 hr.			1000° C., 1 hr.		700° C., 3 hr.			1000° C., 1 hr.		700° C., 3 hr.		1000° C., 1 hr.		
hkl	d, Å	I.	d, Å	I.	hkl	d, Å	I.	d, Å	I.	d, Å	I.	hkl	d, Å	I.
111	5·82	2	5·87	2				3·87	5				4·0	1
			4·67	2	113	3·10	1	3·12	1				3·67	1
			3·59	1	222	2·98	10	2·98	10				3·18	10
113	3·08	1	3·09	2				2·74	7			111	3·11	8
			3·01	4	004	2·59	3	2·59	4	2·93	1vb		2·93	3
222	2·93	10	2·95	10	115/333	2·01	1	1·99	1			002	2·71	5
			2·90	2				1·94	4				2·53	2b
			2·74	1b	044	1·83	7	1·83	10	2·15	1vb		2·15	1b
004	2·55	6	2·56	6				1·74	3				1·98	1
			2·44	1				1·59	5			022	1·92	9
			2·20	1	226	1·56	7	1·56	10	1·89	1vb			
			2·16	2	444	1·49	3	1·50	3				1·86	3
			2·06	1				1·376	3				1·77	2
115/333	1·98	1	1·98	1	355/137	1·348	1vb	1·358	1	1·64	1vb	113	1·64	9
			1·90	4b	008	1·294	2vb	1·296	4			222	1·57	2
			1·85	1	266	1·189	4b	1·190	7				1·52	2
044	1·81	8	1·81	9	048	1·153	4b	1·160	7				1·46	2b
			1·74	4				1·125	1b			004	1·361	4
			1·70	2				1·080	1b			133	1·247	5
			1·58	1b	448	1·060	4b	1·060	7b			024	1·214	5
226	1·55	8	1·55	9				1·041	7b			224	1·110	4
444	1·48	3	1·49	4	666	1·000	4b	1·000	7vb			115/333	1·047	4
			1·46	3								044	0·963	2
355/137			1·344	1b								135	0·920	4
												006/244		2
008	1·283	2b	1·288	4								026		4
266	1·179	4b	1·183	6								335		2
048	1·149	4b	1·152	6										
448	1·049	3b	1·053	5										
666	0·989	3b	0·994	5										

the powder patterns might be ascribed to varying proportions of the phases.

Berman (1955) pointed out that as allanite melts or decomposes at a relatively low temperature it would not be advisable to heat allanite above the temperature of 850° C., if reliable X-ray patterns corresponding to simple recrystallization are to be obtained. For identification purposes what seems to be important is to obtain a characteristic diffraction pattern for a mineral species, and not necessarily from its original structure. Consequently, melting during the heat treatment will not be any disadvantage provided that a good crystallization is obtained after cooling. Ueda and Korekawa (1955) showed that some allanites melt at 1400° C. and that good diffraction patterns could be obtained after cooling.

For comparison with the results obtained by Berman we have also taken X-ray powder photographs of five of these allanites heated in air to 800° C. for ½ hour. The results obtained are similar to those at 700° C., and do not include any powder patterns suitable for identification purposes.

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