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RE-EXAMINATION OF "STRUVERITE" FROM SALAK NORTH, MALAYA*

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ABSTRACT

Critical re-examination of the original "struverite" from Salak North, Malaya, shows that the samples contain much ilmenite and some cassiterite. As a result, the analysis given by Crook and Johnstone in their description of the mineral in 1912 (1) indicates some contamination by ilmenite and cassiterite, and the subsequent ASTM *x*-ray powder data card for "struverite" (2-1354) gives a mixed pattern of "struverite" and ilmenite. Furthermore, due probably to the difficulty of analysing Ta/Nb at that time, an incorrect, and very high, value of tantalum was quoted in the original analysis. The mineral is actually richer in niobium than in tantalum and should properly be termed 'ilmenorutile' and not "struverite." Neither of these terms is favoured by the present writer who prefers the terms "*niobian-rutile*" and "*tantalian-rutile*" respectively.

Using more sensitive methods of magnetic separation together with *x*-ray powder and polished section studies the mineral is found to be homogeneous, consisting essentially of titanium, niobium, tantalum, and iron. A definite, but complex, relationship between the composition and specific gravity and mass magnetic susceptibility is indicated. *X*-ray powder patterns of the most magnetic material give an apparent bi-rutile structure, which can be seen to develop from a rutile structure in the less magnetic material. As such the mineral is intermediate between rutile and tapiolite, an isomorphous series postulated by Prior in 1908 (2).

INTRODUCTION

This paper is part of a general investigation into the relationship of rutile (TiO_2) and the tetragonal tapiolite-mossite series ($Fe(Ta,Nb)_2O_6$). Methods of study include electromagnetic separation, chemical analyses, certain physical and optical determinations, examination of polished sections, and *x*-ray powder work.

The first discovery of "struverite" (Ta/Nb-rutile) in Malaya was made in 1909 and was described by Crook and Johnstone in a paper published in 1912 (1). It occurred as alluvial grains obtained from a tin-mining lease near Salak North in Perak. The only material available to the writer was that of three concentrates, retained in the Central Reference Collection of the Geological Survey of Malaya and registered as GS: 3140, 3141 and 4893, labelled "struverite." The total weight of each concentrate was only a few grammes.

PREPARATION OF THE MATERIAL

The three concentrates available to the writer were electromagnetically separated on a Frantz Isodynamic separator. Using a side slope of 25° and a forward slope of 15°, each sample was separated by passing

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TABLE 1. ELECTROMAGNETIC DISTRIBUTION OF THE Ta/Nb RUTILE

Fraction	Magnetic at	Sample No.			
		3140		4893	
		Wt. (gm)	%	Wt. (gm)	%
d	0.6 amps.	—	0	—	0
e	0.7 amps.	1.35	83.9	0.57	71.3
f	0.8 amps.	0.07	4.3	0.02	2.5
g	0.9 amps.	0.09	5.6	0.10	12.5
h	1.0 amps.	0.10	6.2	0.11	13.7
		1.61	100.0	0.80	100.0

Chemical assays for $(Ta/Nb)_2O_5$ on fractions (d) and the more magnetic fractions gave a maximum value of 0.27%, an average figure for Malayan ilmenites.

through field intensities induced by current increments of 0.1 amp. Examination of the various fractions under the microscope, combined with chemical analyses for $(Ta/Nb)_2O_5$ and zinc-dish treatment for the detection of any cassiterite, gave the following approximate compositions:

	3140	3141	4893
Ta/Nb-rutile	1.61 gm.	0.25 gm.	0.80 gm.
Ilmenite	3.26	7.03	0.55
Cassiterite (some magnetic)	1.39	0.45	2.82
Xenotime and monazite	—	1.15	—
Tourmaline	—	—	0.08
	6.26 gm.	8.88 gm.	4.25 gm.

Because of the small amount of Ta/Nb-rutile in sample 3141 no further work was carried out on it. The electromagnetic distribution of the Ta/Nb-rutile in samples 3140 and 4893 is given in Table 1.

CHEMICAL COMPOSITION

Crook and Johnstone (1912, p. 226) gave the following quantitative analysis of the mineral:

TiO_2	45.74%
Ta_2O_5	35.96
Nb_2O_5	6.90
FeO	8.27
MnO	trace
SnO_2	2.67
SiO_2	0.20
H_2O at 105° C.	0.08
H_2O above 105° C.	0.42
	100.24%

They state that the SiO_2 occurs as free quartz and indicate that the SnO_2 is an impurity. The presence of magnetic cassiterite detected in the re-examined concentrate strongly supports their assumption. The mineral is therefore an oxide of iron, titanium, tantalum, and niobium. Crook and Johnstone state that "there is apparently no reasonable alternative to the view suggested by Prior that 'struverite' is a homogeneous isomorphous mixture, and it is to be regarded as consisting essentially of a solid solution of tapiolite in rutile." Ignoring the SiO_2, SnO_2 and H_2O they recalculate the analysis to give the following ratio of the two molecules:

TiO_2	41.39%
$Fe(TaNb)_2O_6$	58.61

All evidence supports their view that the "struverite" is a "homogeneous isomorphous mixture," but one that is a continuous series rather than a mixture of the rutile and tapiolite molecules.

The structural formula for Ta/Nb-rutile is given by Dana (3) as $Fe_x^{2}(Ta,Nb)_{2x}Ti_{1-3x}O_2$, with the condition that x must not exceed about 0.2. This can be written as $Fe_x^{2}(Ta,Nb)_{2x}Ti_{60-3x}O_{120}$, with x not exceeding about 12.

Recalculating the original analysis by Crook and Johnstone on a basis of 120 oxygens in the unit cell, the number of cations in the unit cell is:

38.21 Ti
10.86 Ta
3.49 Nb
7.69 Fe
60.25

This gives a structural formula of $Fe_{7.69}(Ta,Nb)_{14.35}Ti_{38.21}O_{120}$ which fits almost perfectly, the slight excess of Fe being attributable to some iron oxide or a little ilmenite.

The high tantalum figure was a point of great interest, as no other tantalum/niobium oxide, except a euxenite, has been found in Malaya with Ta exceeding Nb. Because of the small amount of material available to the present writer it was not possible to carry out a complete analysis by wet chemical methods. A partial analysis of fraction (e), the most magnetic material, gave:

	3140(*e*)	4893(*e*)
% Nb_2O_5	17.80	15.92±0.24
% Ta_2O_5	14.18±0.18	13.90±0.26

The difference between the two results is striking. There would appear to be two possible explanations. Either the original analysis is correct

and the present material is not representative of the original sample, or the original analysis is incorrect due to the great difficulty in separating the titanium, tantalum and niobium oxides at the beginning of the century. All available evidence indicates the latter possibility. It is also possible that the TiO_2 figure is too low and that the original sample contained much more ilmenite, not mentioned by Crook and Johnstone, but making up the bulk of the present material, and that the close agreement of the structural formula is fortuitous.

According to the original analysis the tantalum/niobium ratio is 81:26 and the mineral would rightly be named "struverite" (4). The recent analyses give tantalum/niobium ratios of 32:67 and 31:60 respectively. Thus the bulk, if not all, of the mineral is in fact "ilmenorutile" (4). Neither of these terms is favoured by the present writer who prefers the terms "*tantalian-rutile*" (with the presence of a variable, but subordinate, amount of niobium implied) and "*niobian-rutile*" (with the presence of a variable, but subordinate, amount of tantalum implied) respectively.

From the relationship

$$\text{mass susceptibility} = \frac{\text{magnetic susceptibility}}{\text{density}}$$

the less magnetic (i.e. lower mass susceptibility) fractions of the material might be expected to have a higher proportion of tantalum to niobium, due to a higher density. It is thus quite possible that some of the Salak North material may be tantalian-rutile. This indicates the undesirability of having specific names for the two varieties. The term Ta/Nb-rutile covers the entire sample.

Physical and Optical Characters

The mineral is black, with a metallic lustre which is usually bright but sometimes dull. The grains average 2 mm. in length and 1 mm. in width. They are usually rounded, less often sub-rounded with four or five faces showing when a prismatic habit can usually be distinguished.

Crook and Johnstone reported that the particles, when crushed, are not opaque but are either brownish-yellow (O-ray) or strongly pleochroic in brownish-yellow and dull bluish-green (E-ray). The present writer considers the brownish-yellow and the dull bluish-green to be more of an orange-brown and dark bottle-green respectively. A difference was also found in the degree of opacity according to the mass magnetic susceptibility of the mineral. In the weakly magnetic material (fraction *g*) the translucency can be seen quite easily under fairly low power. With the most magnetic material (fraction *e*), however, this can be seen only under

high-power and then usually on the edge of the fragment only. Similarly the colour of the crushed powder varies from dark chocolate-brown in (*e*) to light purple-brown in (*h*), the latter closely resembling the fawn powder of pure rutile.

It was not possible to obtain an interference figure because the grains had to be crushed extremely fine to achieve translucency, but the mineral is known to be uniaxial (from *x*-ray data) and positive in sign (because the E-ray is the slower of the two). No attempt was made to obtain the refractive indices of the mineral.

Using a Berman density balance the specific gravity of three single grains of the most magnetic material (fraction *e*) was obtained. The resulting values of 4.876, 4.968 and 5.138 show considerable variation and indicate a wide range of chemical composition. A wide range of values within a magnetic fraction is to be expected, for even if there is a relationship between the chemical composition, mass magnetic susceptibility and density, it could not be a simple one. For any given composition in which the ratio of Ta to Nb remains constant the density and mass magnetic susceptibility of the mineral would be expected to increase in direct relationship to an increase in total Ta/Nb (and therefore Fe″) content. For any given composition in which the total Ta+Nb (and therefore Fe″) content remains constant, the density will be expected to behave as for the columbite-tantalite series, increasing as the Ta increases and decreasing as the Nb increases. The mass magnetic susceptibility, however, will be the reverse because of the higher density of the tantalum-high end. Thus a given density or mass magnetic susceptibility value could be due either to a high tantalum-high titanium or to a high niobium-low titanium composition.

Examination of Polished Sections

An examination in reflected light of polished grains of fractions (*e*) to (*h*) of sample 3140 gave the following data:

Homogeneity

The mineral is perfectly homogeneous. Both the weakly and strongly magnetic material, because of twinning, exhibit irregular small patches, sometimes constituting nearly half the area of the grain. These slightly darker patches are barely visible in plane polarized light, but with crossed nicols (Fig. 1) they are prominent because of the strong anisotropism of the mineral. These patches of twinning indicate different degrees of reflectivity according to the crystallographic orientation, and their irregularity would seem to indicate a random distribution of the replacing ions.

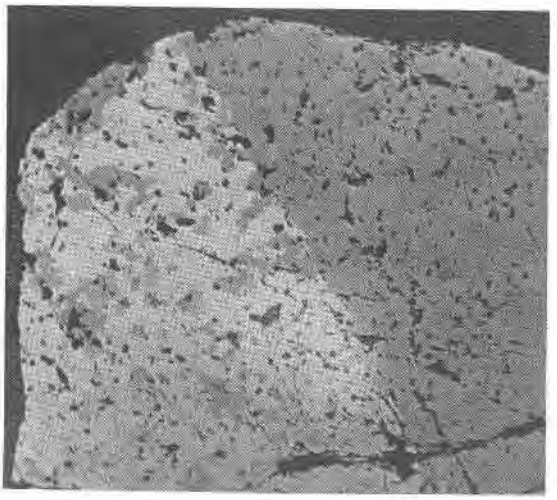

FIG. 1. Single grain of Ta/Nb rutile (sample 3140e) showing irregular patches due to different orientation. Crossed nicols. ×100.

Hardness

Using a micro-hardness tester on a Vickers Projection microscope, two series of tests were made with weights of 50 gm. and 100 gm. respectively. For each series, six readings were taken for each of three fractions, three for the main bulk of the mineral and three for the twinning "blebs." The three fractions included the most magnetic and the least magnetic material from one concentrate. Comparative tests were made on samples of pure rutile and columbite. The results are given in Table 2.

TABLE 2. HARDNESS TESTS

Sample	Weight applied in grams			
	50		100	
	*d**	HV	*d**	HV
3140*e*	4.0	927	6	824
3140*g*	4.0	927	—	—
3140*h*	4.0	927	5.5	981
Rutile	3.5	1211	5.5	981
Columbite	4.25	821	6.5	702

d=mean diameter of indentation (1 unit=2.5 microns).
HV=Vickers hardness number.
* Mean of six readings. In samples 3140*e*, *g* and *h* the six readings were constant

With a 50 gm. load the nioban-rutile is intermediate between rutile and columbite and shows no variation. A 100 gm. load, which is probably more accurate because of the larger indentation, indicates a decrease, from a value similar to rutile to a value approaching columbite, with an increase in mass magnetic susceptibility. The readings are insufficient, however, to give this a statistical basis.

Other properties

The mineral has C/D reflectivity (5). Under crossed nicols it shows strong anisotropism with two positions, at 90°, which are light yellow-grey and dark grey, there being four maxima during each complete revolution. In addition to the random patches mentioned previously the grains show normal twinning (Fig. 1).

The internal reflection varies from a deep red, easily visible, in fraction (*g*) to none in fraction (*e*).

X-Ray Powder Data

The ASTM *x*-ray powder data card (2-1353) for "ilmenorutile," from the Ilmen Mts., Ural, gives a perfect rutile pattern with a slight increase in the *d*-spacings over the values for pure rutile. The ASTM powder data card (2-1354) for "struverite" is of material taken from Salak North, Malaya. The pattern is a very complex one, but closer examination shows it to be a mixture of rutile and ilmenite patterns. This is not surprising considering the high percentage of unsuspected ilmenite present in the original sample, which was thought to be pure. The relevant data are reproduced in Table 3.

The writer obtained powder photographs of the various magnetic fractions of both samples. Only one grain was used in each case. The mineral was found to exhibit two distinct patterns. The less magnetic material gave a rutile pattern with increased *d*-spacings and unit cell dimensions and a few extra faint lines due to a superlattice. The most important of these extra lines, which become stronger with an increase in magnetism, is at $d = 2.9^{+}$. This is the strongest line in the columbite pattern, but it can be indexed as (001) in the rutile pattern. One of these patterns (3140*g*) is given in Table 4.

The more strongly magnetic material gave a complex pattern. Results from two such grains (3140*e* and 4893*e*) are given in Table 4. This appears to be a combination of rutile and columbite. Alternatively it can be indexed as a single pattern of a tetragonal mineral with a_0 slightly larger than for rutile and c_0 approximately double. The mineral is homogeneous, so that it would appear to possess a structure which is either a mixture of a rutile and a columbite phase or a *bi-rutile* one. The

writer is of the opinion that the latter is the correct interpretation. As such the mineral is structurally intermediate between rutile and tapiolite, with its tri-rutile structure. Plotted against the *x*-ray powder data of rutile (4-0551) and tapiolite (2-1341), the two bi-rutile patterns given fit perfectly. They also indicate some inaccuracies in the indexing of the tapiolite. Part of the plot is given in Fig. 2. Some extra lines, due to a tri-rutile super-lattice, are present in both patterns.

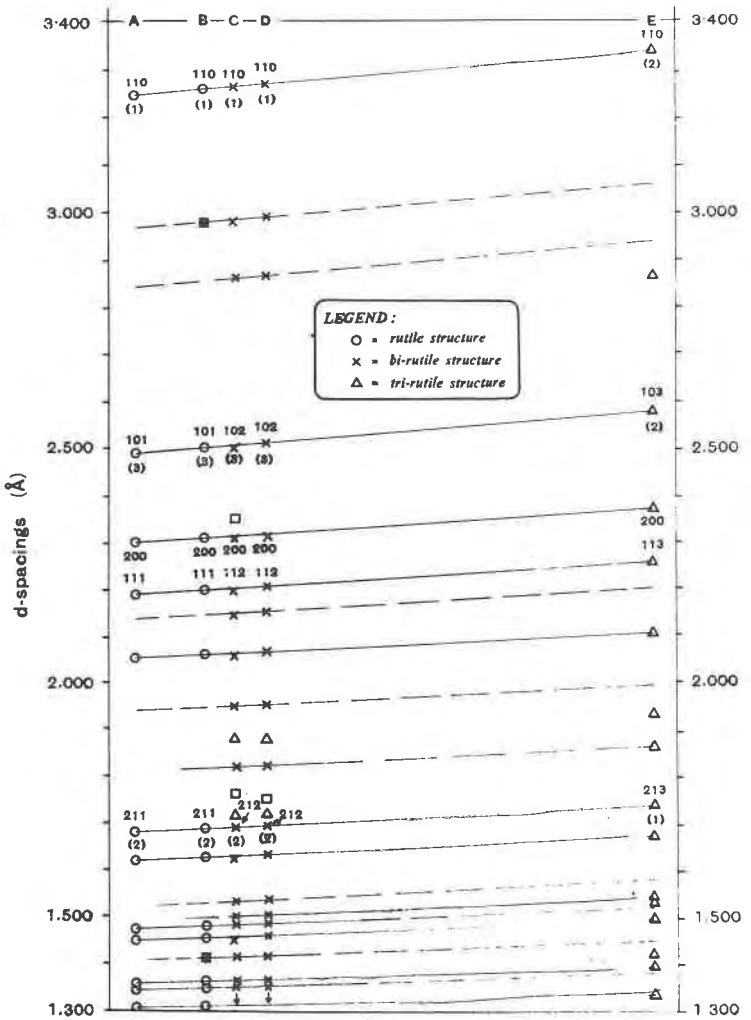

FIG. 2. Plot of a portion of the *x*-ray powder data to show the relationship of the rutile, bi-rutile and tri-rutile structures. For explanation, see next page.

Fig. 2 represents d spacings (for the range $d=3.400$ to 1.300 Å) which have been indexed to indicate the relationship between the rutile, tri-rutile and the proposed bi-rutile structures, as follows:

A Rutile ASTM-4-0551	B[a] Sample 3140*g*	C Sample 4893*e*	D Sample 3140*e*	E Tapiolite ASTM-2-1341
110	110	110	110	110
—	001[b]	002	002	—
—	—	111	111	N.I.[c]
101	101	102	102	103
—	—	N.I.[c]	—	—
200	200	200	200	200
111	111	112	112	113
—	—	201	n.m.[d]	—
210	210	210	210	202[e]
—	—	211	n.m.	—
—	—	t.s.	t.s.	212
—	—	202, 103	202, 103	N.I.[f]
—	—	N.I.	N.I.	—
211	211	t.s.[g] 212, 113	t.s. 212, 113	213
220	220	220	220	220
—	—	300	n.m.	—
—	—	203	n.m.	301, 214
002	002	004	004	006[h]
310	310	310	310	310, 311[j]
—	102[b]	104	n.m.	—
—	—	—	—	312
301	301	302	n.m.	303, 116[k]
112	112	114	114	—
311	311	↓	↓	313

(*a*) Values for C should be plotted coincident with B, but are separated on the diagram for clarity.

(*b*) For line B values of $d=2.981$ and $d=1.4185$ which have been indexed on the rutile lattice as 001 and 102 respectively can alternatively be due to the bi-rutile super-lattice.

(*c*) N.I. means "not indexed."

(*d*) n.m. means that a line is present on the film but it is not measurable.

(*e*) The value of $d=2.11$, indexed in E as 202, can also be indexed as 210.

(*f*) The value of $d=1.86$, unindexed in E, can be indexed as 203.

(*g*) t.s. means lines due to a tri-rutile super-lattice.

(*h*) The value for 006, given in E as 1.546, should be 1.5351.

(*j*) The indexing of $d=1.505$ as 311, in E, is incorrect. An index of 311 gives $d=1.4810$.

(*k*) The indexing of $d=1.408$ as 116, in E, is incorrect. An index of 116 gives $d=1.3960$.

Line Intensities: The three strongest lines of each pattern are shown in brackets beneath the appropriate d values.

TABLE 3. ASTM *X*-RAY DATA

'Ilmenorutile' 2-1353		'Struverite' 2-1354		Rutile 4-0551		Ilmenite 3-0781	
d Å	I	*d* Å	I	*d* Å	I	*d* Å	I
—	—	3.87	20	—	—	3.73	50
—	—	3.59	40	—	—	—	—
3.27	70	3.25	80	3.245	100	—	—
—	—	2.76	40	—	—	2.74	100
2.50	50	2.50	70	2.489	41	2.54	85
2.31	10	2.32	40	2.297	7	—	—
2.19	10	2.20	40	2.188	22	2.23	70
—	—	2.07	20	2.054	9	—	—
—	—	1.88	40	—	—	1.86	85
1.70	100	1.70	100	1.687	50	1.72	100
1.63	50	1.64	60	1.624	16	1.63	50
—	—	1.51	20	—	—	1.50	85
1.48	10	1.49	40	1.480	8	—	—
—	—	1.47	50	—	—	1.47	85
1.46	30	—	—	1.453	6	—	—
1.37	30	1.37	70	1.360	16	—	—
1.36	10	1.36	60	1.347	7	1.34	70
—	—	—	—	1.305	1	—	—
—	—	1.26	40	1.243	3	1.27	60
—	—	—	—	1.200	1	1.20	30
—	—	1.18	70	1.1700	4	1.18	60
—	—	1.16	60	1.1485	4	1.15	70
—	—	—	—	1.1329	1	—	—
—	—	1.11	70	1.0933	4	1.12	70
—	—	1.09	60	1.0827	4	—	—
1.05	50	1.05	80? 70	1.0424	5	1.07	70
—	—	1.04	60	1.0361	4	and	
—	—	—	—	1.0273	3	six more lines to	
—	—	—	—	.9642	2	0.913	
—	—	—	—	.9071	3		
—	—	—	—	.9007	3		
.895	30	—	—	.8892	5		
.881	30	—	—	.8773	6		

There appears to be no direct relationship between the position on the plot and the Ta/Nb content. The bi-rutile and tri-rutile patterns seem to appear suddenly at certain values. What these values are is not known, but, from the structural formula, the values of $x=10$ and $x=15$ (i.e. $Fe_{10}(TaNb)_{20}Ti_{30}$ and $Fe_{15}(TaNb)_{30}Ti_{15}$) give a bi-rutile pattern of $(Fe(TaNb))_{30}Ti_{30}$ and $(FeTi)_{30}(TaNb)_{30}$ respectively. Whether the change is

TABLE 4. *X*-RAY POWDER DATA FOR LESS MAGNETIC (3140*g*) AND MORE MAGNETIC FRACTIONS (3140*e* AND 4893*e*). CuKα=1.5417 Å; CAMERA DIAMETER 114.83 MM. A SINGLE GRAIN WAS USED IN EACH CASE

hkl	3140 *g*			*hkl*	3140 *e*			4893 *e*		
	d (Å) (meas.)	*d* (Å) (calc.)	I (visual)		*d* (Å) (meas.)	*d* (Å) (calc.)	I (visual)	*d* (Å) (meas.)	*d* (Å) (calc.)	I (visual)
—	—	—	—	001	5.903	5.973	<1	5.903	5.952	<1
—	—	—	—	100	4.603	4.623	<1	4.589	4.607	<1
—	—	—	—	101	3.627	3.657	4	3.625	3.642	2
110	3.233	3.257	10	110	3.258	3.269	10	3.249	3.258	10
001[a]	2.951	2.981	<1	002	2.944	2.988	8	2.956	2.976	8
—	—	—	—	111	2.8425	2.8679	<1	2.8525	2.8579	<1
101	2.4815	2.5033	8	102	2.5000	2.5098	8	2.4905	2.4997	8
—	—	—	—	N.I.[c]	—	—	—	2.3538	—	<1
200	2.2946	2.3030	1	200	2.3048	2.3115	1	2.2963	2.3035	1
111	2.1900	2.1996	4	112	2.1918	2.2056	3	2.1919	2.1969	3
—	—	—	—	201	↓	2.1563	<1	2.1488	2.1483	<1
210	2.0512	2.0599	1	210	2.0675	2.0675	1	2.0621	2.0604	<1
—	—	—	—	211	↓	1.9539	<1	1.9463	1.9471	<1
—	—	—	—	N.I.[d]	1.8823	—	<1	1.8834	—	<1
—	—	—	—	202, 103	1.8119	1.8280 1.8294	<1	1.8196	1.8217 1.8224	<1
—	—	—	—	N.I.	1.7586	—	<1	1.7640	—	<1
—	—	—	—	N.I.[e]	1.7243	—	<1	1.7220	—	1
—	—	—	—	N.I.	—	—	—	1.7087	—	<1
211	1.6873	1.6946	9	212, 113	1.6975	1.7002 1.7009	9	1.6908	1.6941 1.6944	9
220	1.6268	1.6287	1	220	1.6335	1.6347	4	1.6298	1.6291	2
—	—	—	—	300	↓	1.5410	<1	1.5354	1.5357	<1
—	—	—	—	203	↓	1.5088	<1	1.5061	1.5033	<1
002	1.4807	1.4911	<1	004	1.4940	1.4942	1	1.4841	1.4880	<1
310	1.4540	1.4567	<1	310	1.4621	1.4620	2	1.4570	1.4570	1
102[b]	1.4234	1.4185	<1	104	↓	1.4216	<1	1.4133	1.4160	<1
301	1.3606	1.3651	2	302	—	1.3696	—	1.3669	1.3646	3
112	1.3479	1.3555	<1	114	1.3609	1.3589	4	1.3519	1.3534	1
311	1.3129	1.3089	<1	⋮	↓	↓		↓	↓	
202	1.2468	1.2516	<1	↓						
212	1.2138	1.2077	<1							
321	1.1729	1.1743	<1	Arrows indicate the presence of one or more lines too faint and						
400	1.1505	1.1515	<1	diffuse to measure accurately.						
222	1.0965	1.0995	<1							
330	1.0857	1.0856	<1							
411	1.0469	1.0461	<1	412	1.0514	1.0497	2			
312	1.0409	1.0418	<1	314	1.0459	1.0450	1½			
↓	↓	↓								
402	.9107	.9114	<1	⋮	↓	↓				
510	.9029	.9033	<1	↓						
213	.8939	.8952	<1	216	.8965	.8973	<1			
N.I.	.8902	—	<1	—	—	—	—			
431	.8819	.8802	<1	432	.8841	.8833	1½			
332	.8784	.8777	<1	⋮	↓	↓				
422, 223	.8479	.8475 .8484	<1	↓						
303	.8331	.8344	<1	306	.8363	.8364	<1			
521	.8234	.8222	<1	522	.8270	.8251	<1			
	$a_0=b_0=4.606$ Å $c_0=2.982$ Å $a:b:c=1:1:0.6474$				$a_0=b_0=4.623$ Å $c_0=5.976$ Å $a:b:c=1:1:1.2927$ S.G. (measured): 4.968			$a_0=b_0=4.607$ Å $c_0=5.950$ Å $a:b:c=1:1:1.2915$ S.G. (measured): 4.876		

[a] Alternatively is bi-rutile super-lattice (002).
[b] Alternatively is bi-rutile super-lattice (104).
[c] Tri-rutile super-lattice (200).
[d] Tri-rutile super-lattice (212/203).
[e] Tri-rutile super-lattice (213). The strongest line.

made at these values or below and above them cannot be stated until complete analyses have been carried out on single grains for which the *x*-ray patterns are known.

A hypothetical consideration of the possible structural changes from the tetragonal (rutile) form of TiO_2 to the tetragonal form of $FeTa_2O_6$

(Nb being omitted for simplicity) is given schematically in Fig. 3. This disallows any limiting value on x, which theoretically can range from zero ($Ti_{60}O_{120}$) to 20 ($Fe_{20}Ta_{40}O_{120}$), in the structural formula. Taking six unit cells (in the c_0 direction) of rutile there are six Ti ions equally spaced at a value of $c_0 = r$. As Fe″ and Ta (ignoring Nb, which would go with the Ta, for simplicity) enter the formula the ions substitute randomly for Ti giving six TiTaFe positions with no change in c_0, but a super-lattice begins to form. At a certain value of Ti, which is probably above but certainly at 30 (i.e. $x = 10$ or less in the structural formula) the structure changes to a bi-rutile one, the c_0 repeat distance being $2r$ and the ion

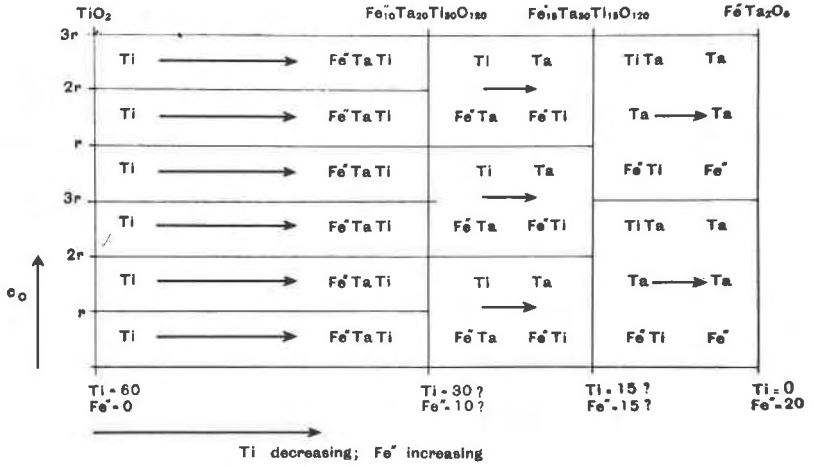

FIG. 3. Hypothetical structural changes.

positions being taken by Ti and FeTa. Between the values of Ti = 30 and 15 (i.e. $x = 10$ & 15) Ti and Ta change places and at Ti = 15 the ion positions are occupied by FeTi and Ta. As the Ti decreases some of the Ta occupies the FeTi positions and a super-lattice begins to form until, at a certain value of Ti a tri-rutile structure obtains giving a repeat distance of $3r$. Whether the change from a bi-rutile to a tri-rutile structure does occur in nature can only be shown by studying low-titanium material, but the presence of a tapiolite super-lattice in the two bi-rutile patterns given indicates that it does.

CONCLUSION

Tantalian- and niobian-rutile (i.e. "struverite" and "ilmeno-rutile") have long been regarded as members of an isomorphous rutile-tapiolite series though proof of such a series is still lacking. The niobian-rutile from

Salak North is a mineral of variable composition, structurally and chemically intermediate between rutile and tapiolite and with intermediate optical and physical properties. It establishes the presence of an isomorphous series for the rutile end, the members varying from a Ta/Nb-free mineral with a rutile structure to a Ta/Nb-rich mineral possibly with a bi-rutile structure. Whether the series at the Ti-free end is complete is not known. There appears to be no valid reason for doubting its existence though it can be proved only by a continued search for its members.

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